Monitoring the Progress of Retting in Bast Fibre Crops

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ABSTRACT

Retting is the crucial process that dissociates fibres from the stem of bast plants; the inter-fibre matrix is digested by the action of microbial enzymes. Effective retting enables valuable, undamaged and uncontaminated fibres to be extracted from the stem using relatively simple mechanical processing. The progress of retting is largely assessed using traditional, subjective assessments. No reliable, objective method has previously been devised to effectively monitor the progression of retting in the field.

This study investigated three different mechanical test methods that enabled rapid, reliable, and reproducible monitoring of the retting process in bast crops. The peel test measured the work done in dissociating fibre bundles from the stem during retting. The work to peel decreased as the inter-cellular matrix was digested and this was used to indirectly monitor the progress of retting. The effects of peeling angle and sample moisture content were also investigated. The tear test measured the force applied when fibre bundles were separated from each other. Although results were more variable, the general trend was for tear force to decrease, indicating the progression of retting. A novel laboratory decorticator was devised, which enabled a reproducible amount of decortication work to be administered to individual stem samples. A profiled metal roller was rolled down an inclined plane with a congruous profile and over the samples. The stems were decorticated to produce ribbons of fibre; loose but entangled shive was removed using a low velocity airstream. The proportion of shive removed by each subsequent decortication pass was calculated and related to the ease of decortication, allowing the effect of a range of parameters to be compared. The inclined plane decortication method was validated by decorticating straw that had been enzyme-retted under controlled conditions. Results showed good correlation between the duration of retting and the ease of decortication.

CONTENTS

List of Appendices

List of Figures

List of plates

Chapter 1: Rationale

There is increasing interest in the use of natural fibres in a range of textile markets (technical, medical, household and apparel). Other uses in composites (both structural and non-structural) and building materials (fibre-boards, reinforced materials, lignin-based adhesives etc) are also being developed. Natural fibres have excellent technical qualities and distinct environmental advantages over synthetic alternatives; they are renewable, sustainable and their end products are biodegradable. The potential demand for such fibres is expected to increase dramatically, opening up opportunities in the medium to long-term for growing large hectarages of natural fibre crops across the EU.

Currently, the global textile industry uses some 50 - 60 million tonnes of fibre each year; around half of this total is natural fibres and half is synthetic. The vast majority of the natural fibre consumption is cotton (almost 20 million tonnes), with a relatively small amount being made up of various bast fibres (around 4 million tonnes). The global consumption of all textile fibres is expected to increase dramatically, and that of natural fibres is set to reach around 40 million tonnes per year by 2050, (Koslowski *et al.*, 2002). The global cultivated area of cotton is unlikely to increase significantly and, in any case, further increases would be environmentally undesirable due to the crop's high requirement for pesticide, fertiliser and water. Any increased cotton supply is likely to be limited, coming from more intensive production rather than an increased area under cultivation. With the total textile fibre consumption increasing, and a static cotton supply of around 22 million tonnes per year by 2050 (Koslowski *et al.*, 2002), there will be an increasing demand for alternative fibres. Much of this will be met by an increase in the supply of synthetic fibres of petroleum origin, which is

clearly unsustainable in the long-term. The shortfall between demand for natural fibres and the supply of cotton will open up niche opportunities for other natural fibres (Schenek, 2002). The ultimate fibres (single cell) from crops such as flax and hemp are similar to cotton in some respects and with some refinements they could be used to augment or replace cotton in certain instances. However, unlike cotton (which is an independent, single cell, seed coat fibre) bast fibres form an intimate and integral part of the plant's stem structure; the ultimate fibres are 'cemented' into bundles which themselves are bonded into the stem tissues, making them more difficult to extract and process.

There are other important differences between cotton and bast fibres such as hemp and flax, perhaps the most important being the shape and dimensions of the single fibre cross-section. Cotton fibre has a flattened cross-section with an effective fibre fineness (smallest dimension) of around 5 μm. Cotton fibres always bend in the plane of the larger dimension and so the smaller dimension determines its bending rigidity (an important factor for determining spinnability and prickle factor, both crucial elements of yarn production).

The production of yarn is very complex; put simply, the fibres are held together in the yarn by friction and so the quality and integrity of a yarn is determined by parameters such as the diameter, length, strength, bending properties and surface characteristics of its individual fibres. If bast fibres are to be incorporated into blended yarns with cotton, the fibres have to be compatible with cotton processing and spinning systems. They must be equally free of contaminants and dust, they need to have similar bending properties and surface characteristics, and they need to be of similar length and strength, to ensure that they behave in a similar way to cotton whilst passing through the processing system.

Traditional bast fibre yarns, such as those used in the production of linen (flax fibre), utilise largely intact fibre bundles and this explains some of the characteristics of the resulting materials: their strength, durability and tendency to crease. The fibre bundles of both flax and hemp are more or less cylindrical, with diameters of 50 to 250 μm and lengths of many centimetres. Clearly these are not suitable for blending with cotton without substantial further refinement. Further division of the fibre bundles into ultimate fibres is necessary. The individual ultimate fibre cells of flax have a polygonal cross-section (which is more or less circular) with a notional fibre diameter of around 5 – 30 μm and they have a fibre length of around 5 – 75 mm (typically 20 – 25 mm). A proportion of such fibres are suitable for blending with cotton to produce valuable yarns, increasing the value of the fibre by around 500%.

A programme of research by the Textile Engineering and Materials (TEAM) research group at De Montfort University investigated the potential use of bast fibres in high value textile end uses; particular interest was centred on flax but hemp and nettle fibres were also considered. The aim was to produce flax fibres in the UK that were suitable for inclusion with cotton in blended yarns.

Traditional bast fibre crop production and processing techniques have not targeted the production of cotton-like fibres. Most flax crops are produced for the linen industry where demand is determined mostly by the fashion trade, a relatively small, niche market. A small amount of fibre has been "cottonised" for use in blended yarns, but no method has yet been successful in reliably and economically producing the large quantities of high quality fibre required by cotton spinners. The cotton spinners face a dilemma; short staple length flax fibre

that is compatible with cotton is highly desirable, but cotton spinning is a highly mechanised and finely tuned process, and any move to include a different resource would be very expensive. Before any wholesale changes can be seriously considered there needs to be a continuous and guaranteed supply of suitable raw material. For example, the global cotton industry holds around a year's supply of cotton in stock at all times.

In the past, profitability of EU bast fibre crops has been dependent on the under-pinning production subsidies and as implied by the name, these subsidies were paid regardless of the quality or end use of the crop and provided no incentive for growers to produce a quality product; high quality crops received no more subsidy than poor quality crops. As these subsidies have declined and with no profitable end-use in place, the popularity of bast crops has also declined. Thus, the continuity of supply has proved unreliable. Hemp and flax are particularly suitable for cultivation in Europe, but other bast fibre crops such as *Urtica spp.* could also make a contribution. The cultivation of natural fibre crops in the EU stood at around 15,000 ha for hemp and 95,000 ha for flax in 2001, but cultivation levels for both crops have previously been much higher. The EU is capable of cultivating significantly greater hectarages of natural fibre crops, especially with the imminent membership of several eastern European countries such as the Czech Republic and Hungary. With modified production and processing techniques, penetration into the higher value sectors such as short staple fibre for inclusion in cotton blends, is possible. Typical EU bast fibre crop production techniques have relied on dew-retting. This technique is heavily dependent on the weather conditions after the crop has been cut and laid in swaths on the ground, limiting production to certain geographical locations where the climate is suitable.

The dew-retting process is not easily controlled and typically produces fibres with poor uniformity that are graded to the lowest common denominator and as a consequence, are often of low value. The crop is at serious risk of losing both yield and quality due to adverse weather conditions whilst it is laid on the ground during the retting period.

Thus, if these crops are to successfully and profitably supply bulk resources to the textile industry, the questions of uniformity, quality, reliability and continuity of supply must be addressed. Further reform of the EU Common Agricultural Policy promises the decoupling of subsidies from production for all crops. Under these conditions the implications for fibre production are unclear. The cultivation of traditional high input crops (e.g. wheat) on marginal land will be less attractive once the production subsidy has been withdrawn, making low input (or no input) fibre crops become more attractive. However, in a situation where single farm payments are paid regardless of production, growers will not grow any crop at all unless it is profitable in its own right.

Bast fibre crop investigations carried out at De Montfort University.

Major research projects at De Montfort University, such as FAIR CT-98 9574 (1998) and TEXFLAX (2002) along with a number of smaller projects such as an industry sponsored hemp trial (Fibrenova, 2001) and an East Midlands Development Agency sponsored hemp trial (EMDA, 2002) continue to investigate the cultivation and processing of bast fibres in the UK. In addition, an investigation of the potential of *Urtica dioica* as an alternative source of bast fibre has been planned, funded under the Sustainable Technologies Initiative (STI, 2004). The effects on ultimate (single cell) fibre quality of agronomic factors such as choice of variety, drilling date, plant density, method of crop growth

termination, timing of growth termination, maturity of plant and duration of retting period are being investigated.

The main aim of this research programme is to produce fibres suitable for blending with cotton for use in the higher value textile market sectors. A reliable system of production that reduces the risk of crop yield and quality losses due to adverse weather conditions, which is a major problem during the autumn in northern Europe, is a pre-requisite. A more robust production system promises to improve grower's confidence in bast fibre crops and promote their widespread cultivation with the potential to produce high quality, high value fibres. Quality in this case is determined by fibre cleanliness (level of non-fibre contaminants), fibre fineness (diameter), fibre length and fibre strength of the ultimate single-cell fibres, but it is also indicated by other factors such as bending rigidity, surface properties (e.g. friction) and fibre colour. All of these quality parameters are crucial if the resulting fibre is to be acceptable to the textile industry for blending with cotton, on cotton spinning systems. Over the six years between 1998 and 2003, 12 small-plot field trials with randomised and replicated plot designs and 6 commercial-scale field trials were completed (Plate 1). These were mostly carried out in the UK, using trials sites in three regions: Cornwall/Devon; the east midlands; and the north of England/Scotland. A further trial was carried out in northern Portugal, where autumn-sown flax was compared to traditionally sown spring flax, and exposed to winter temperatures as low as -10° C. Crops were successfully grown and stand-retted in all regions and the potential to produce high fibre quality was confirmed. Samples of flax straw were collected from plots grown in Holland by the Centre for Genetic Resources of the Netherlands (CGN) (Plate 2) as part of their normal seed regeneration activities.

Plate 1 Commercial scale flax trials, Lincolnshire.

Plate 2. Collecting samples of flax straw from the CGN site, Wageningen, NL.

Plate 3. Accession plots in randomised replicated blocks at Huit farm, Leicester.

The 85 accessions in their core collection, which represent the genetic variation within the entire *Linum spp.* seed-bank, were grown in small plots on a site near Wageningen. CGN retained the seed but only utilised a small proportion of the straw, thus a sample of straw from each accession was made available for evaluation in the UK at De Montfort University. These samples were used to compare the fibre quality of the ultimate fibres produced by different accessions grown under similar conditions.

Characterisation of the straw of the 85 accessions, in terms of fibre diameter and fibre diameter distribution, enabled 30 accessions to be chosen for further investigation. However, all 85 accessions were sown since characterisation was not completed before sowing time. Small plots of each accession in the core collection, randomised and replicated in blocks, were grown in the UK in subsequent years for the evaluation of fibre quality produced by the different accessions under UK conditions (Plate 3). Further analysis of these 30 accessions provided a reduced pool of 9 for continued investigation in the following year and this has been reduced to 5 for the final year of the current project.

The task of sample collection for all projects was a time-consuming and complex one. Typically, sampling required 3 - 4 man-days per week. Drying, preparing, labelling and storage of samples was another major task. Only limited fibre quality testing was performed initially, because only small samples of fibres (5 – 10 g) were available. These results enabled an initial screening for promising accessions to be made (fibre cleanliness, fibre diameter and fibre diameter distribution), but larger samples were required for further parameters (fibre length and strength) to be included in the analysis of fibre quality, especially for spinning tests and evaluation of the resulting yarns. Fibre quality evaluations were carried

out by Filartex Ltd of Milan, using standard cotton quality parameters and automated evaluation equipment.

A novel approach to improving the quality of hemp fibre was investigated. In traditionally swathed crops, the longer and more robust stalks of hemp tend to produce a relatively open swath, but often the quantity of foliage included in the swath is substantial. This maintains a high moisture content around the stems and leads to localised rotting which can cause serious crop yield and quality losses if it progresses. Two techniques were investigated; desiccation with Quattro (Syngenta Crop Protection Ltd, glyphosate) followed by stand-retting; and defoliation with Reglone (Syngenta Crop Protection Ltd, diquat) followed by more traditional dew-retting. Each approach sought to modify the moisture levels of the micro-climate around the stems and thus regulate the retting conditions. In each case treatment applications were made using a commercial hydraulic sprayer specifically modified for the purpose (Plate 4).

Hemp crops desiccated with glyphosate would be killed completely and they would proceed to ret as a standing crop prior to harvest. Similarly, defoliation prior to swathing would enable the stems to be laid on the ground in a relatively open, foliage-free swath to take advantage of the more moist conditions on the ground while avoiding the high risk of rotting. Extraction of small samples of fibre from hemp straw and analysis of fibre characteristics to determine fibre quality needed to be developed. The focus of the work on flax and hemp reported here was not the development of processing techniques or the decortication of straw to produce samples for fibre quality analysis, but to develop a method that could monitor the progress of retting so that the effect of various parameters could be quickly and reliably evaluated.

Plate 4. Crop sprayer modified to spray hemp crops, Lincoln.

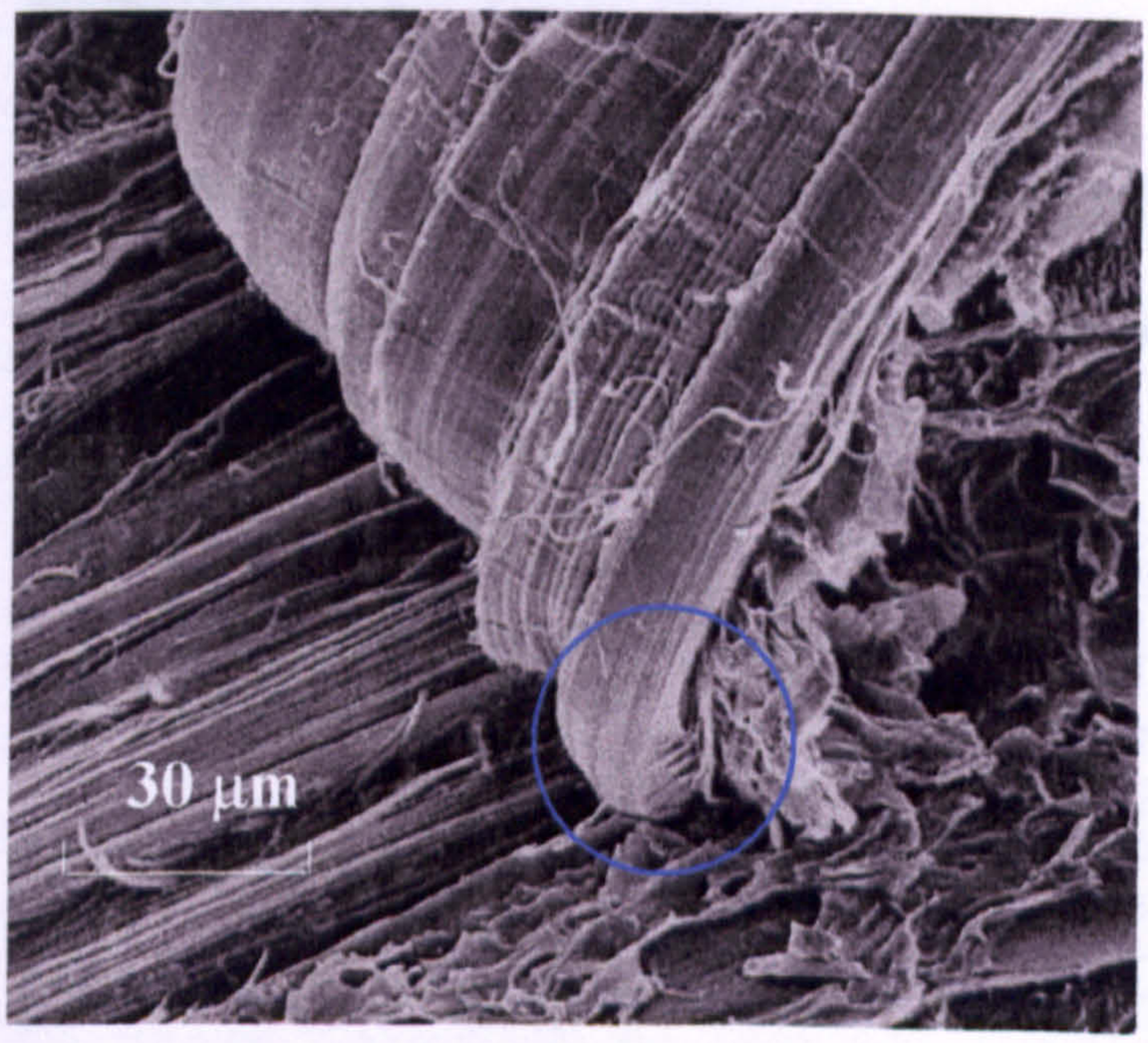


Plate 5. Compression damage on the outer surface of the fibre.

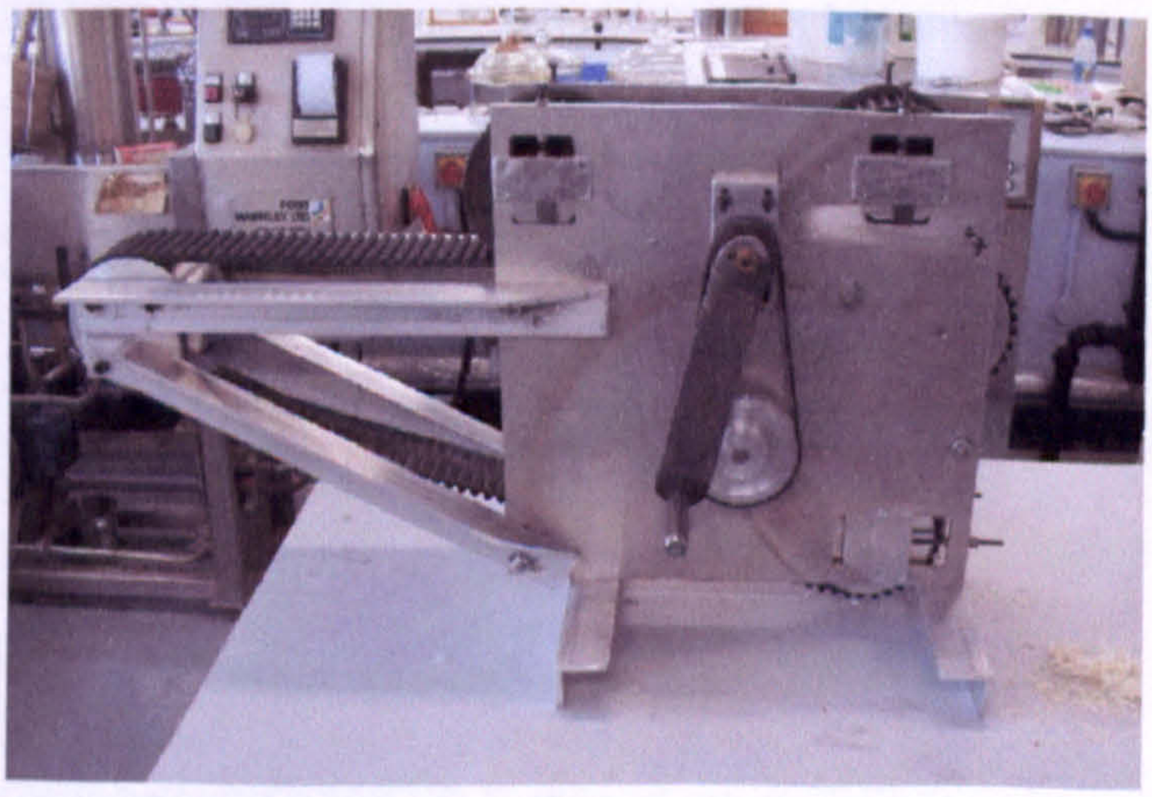


Plate 6. Laboratory-scale prototype decorticator based on a timing belt and drive gear (hand driven).

The techniques investigated produced very small samples of fibre (less than 0.1 g), which were insufficient for fibre quality analysis. Furthermore, in the case of the peel test, especially the modified 180° test, the level of fibre damage caused by the technique may render fibre quality analysis worthless. As the fibres were peeled away from the stem, they were exposed to tension on their inner surface and compression on their outer surface (Plate 5), this is likely to have led to substantial mechanical damage to the fibres themselves and the sample would not be representative of the true fibre quality produced by the treatment being evaluated.

However, in order to extract samples of fibre from both flax and hemp straw, a prototype laboratory-scale decorticator was produced based on the inclined plane decortication test method (Plate 6).

Chapter 2: Introduction

2.1 Background and Aims

Man has utilised natural fibres for thousands of years. There is evidence that bast (stem) fibres from flax and hemp were used by the ancient Egyptians and others to produce a range of textile materials (Van Sumere, 1992). The most crucial stage in fibre production then, as now, was the extraction of the uncontaminated and undamaged fibres from the stem tissue: the cortex to the outer side and the core (shive in flax or hurd in hemp) on the inner side. Since ancient times this has been achieved by harnessing the ability of micro-organisms to partially and selectively digest (ret) the stem tissues, so loosening the fibres and allowing them to be extracted by relatively simple mechanical processing systems. This degradation step of the process, and the overall strategy, has changed little in principle over the millennia, and is still used today, although the detail has been somewhat modified.

When assessing the crop, the degree of retting in straw is usually determined using a series of subjective methods. However, these are mostly used post-harvest by experienced graders, to position a sample of straw into a particular quality band. Thus they cover a very narrow range of "acceptable" qualities, while samples outside these bands are merely graded "unacceptable". When samples are assessed throughout the retting period they will inevitably be graded as "unacceptable" under these criteria for the vast majority of the time, but in order to develop fibre production systems, their progress towards acceptability needs to be understood by the researcher. The same criteria are also used by growers, in many cases with less expertise, to monitor the progress of retting and inform the decision when to harvest, or to predict the eventual fibre quality. However, much

more objective and sensitive techniques are required in order to identify small effects on fibre quality. Using such techniques, researchers may be able to investigate the effects of many individual factors and evaluate their contribution to fibre quality and hence successful fibre production systems.

Bast fibre crops have never been afforded the level of crop research that other major crops such as cereals have enjoyed. Many of the parameters that impact on fibre quality have not been fully evaluated. A better understanding of how the complex interaction between a range of factors affects ultimate fibre quality would allow modern production systems to produce natural fibres with favourable quality characteristics to meet a range of end-user specifications. Since retting is the most crucial step in the production of bast fibres, it is fundamental that techniques are developed that allow researchers to monitor the progress, and effectiveness of, this defining process.

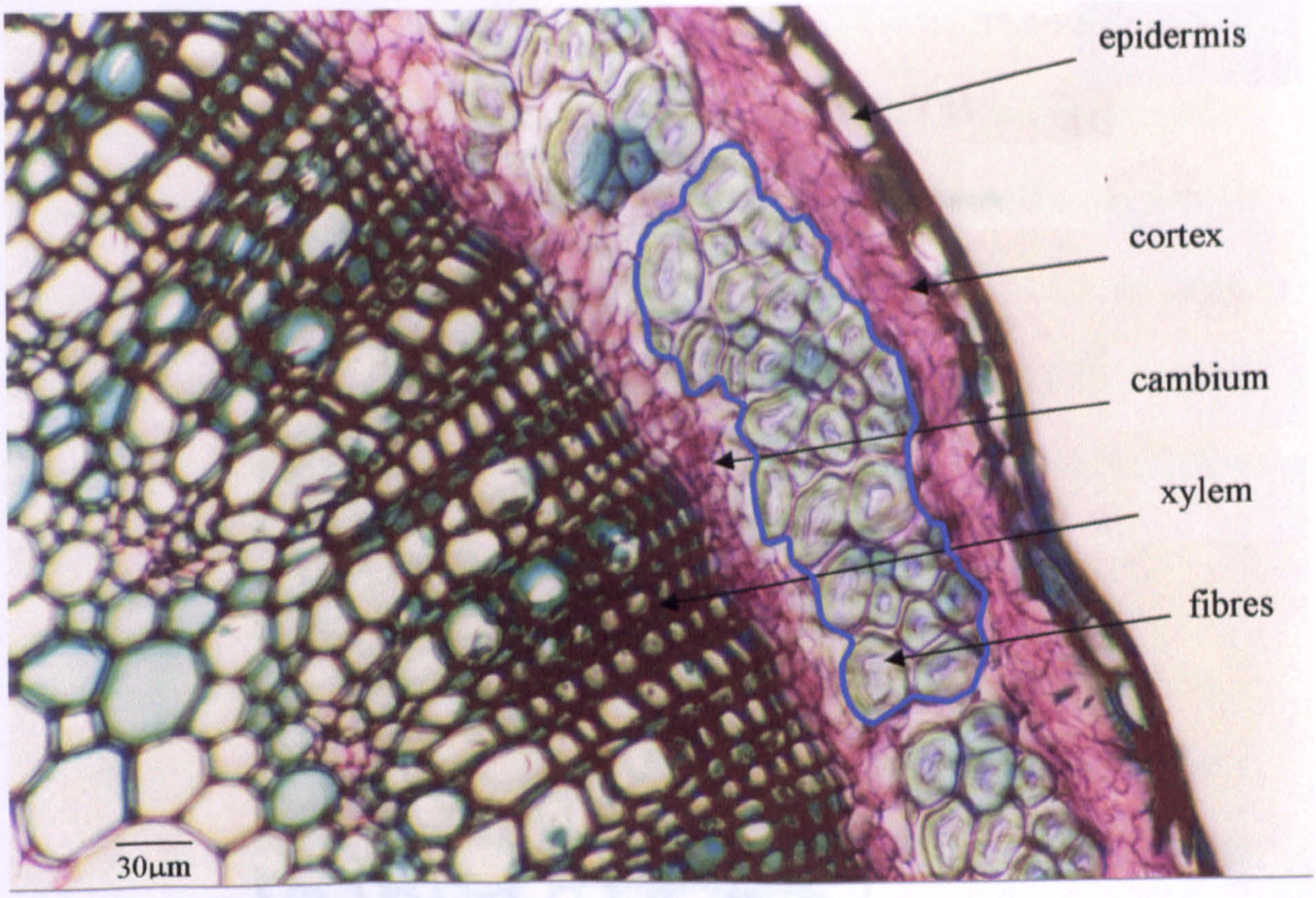
The main aims of this investigation were to reduce the reliance on the traditional subjective assessment methods and develop new objective techniques based on simple mechanical methods. The normal and natural variation in biological systems, even within monoculture cropping situations, necessitates very robust experimental planning and large numbers of samples if recommendations are to be reliable. The assessment techniques need to be rapid, reliable and reproducible, so that the large number of samples generated by the investigations, such as those carried out by TEAM research group, can be assessed with confidence.

If successful, the new techniques should substantially improve the monitoring of retting, enabling researchers to develop enhanced production and processing systems for high quality natural fibres. Eventually, the techniques may be further

developed into tools that could be applied in the field as crop management decision-making aids. An effective, objective system that accurately monitors retting and determines the optimum timing for harvest to meet specifications for particular end uses, could become widely accepted as the industry standard.

2.2 Plant Anatomy and Morphology

Fibre cells may be collenchyma or sclerenchyma type cells. Collenchyma cells differentiate from parenchyma cells and develop thickened primary cell walls, but have no secondary wall. They act as support for growing parts of plants and are alive at maturity eg the stringy tissues in celery stalks. Sclerenchyma cells can develop in any part of a plant and produce very thick cell walls that are often strengthened with lignin (a complex aromatic polymer); they act as supporting elements as the plant matures; these include many important textile fibres. Two forms of sclerenchyma cells develop; sclereids are generally polygonal but variable in shape, while the fibre cells, which develop from phloem cells (responsible for transport of sugars), are generally long and slender (Esau, 1977). Phloem fibres develop in the stems of many plants and some, such as flax and hemp, are utilised as crops to provide sources of natural fibre. In crop situations, the stems of flax usually reach a height of around 1m with a typical mid-stem diameter of 1 - 2 mm, while the stems of hemp typically reach a height of around 3 - 4 m and a diameter of up to 10 mm, with the fibre bundles extending for virtually the entire length of the stem. These cellulose fibres develop from the primary (first formed) phloem cells that are no longer active in transport and they are located in discrete bundles towards the periphery of the stem. (Fig. 1; Plate 7). The surrounding cortex (region of tissue outside the central stele) is not uniform, but typically consists of two to four layers of simple, relatively thin-walled cells



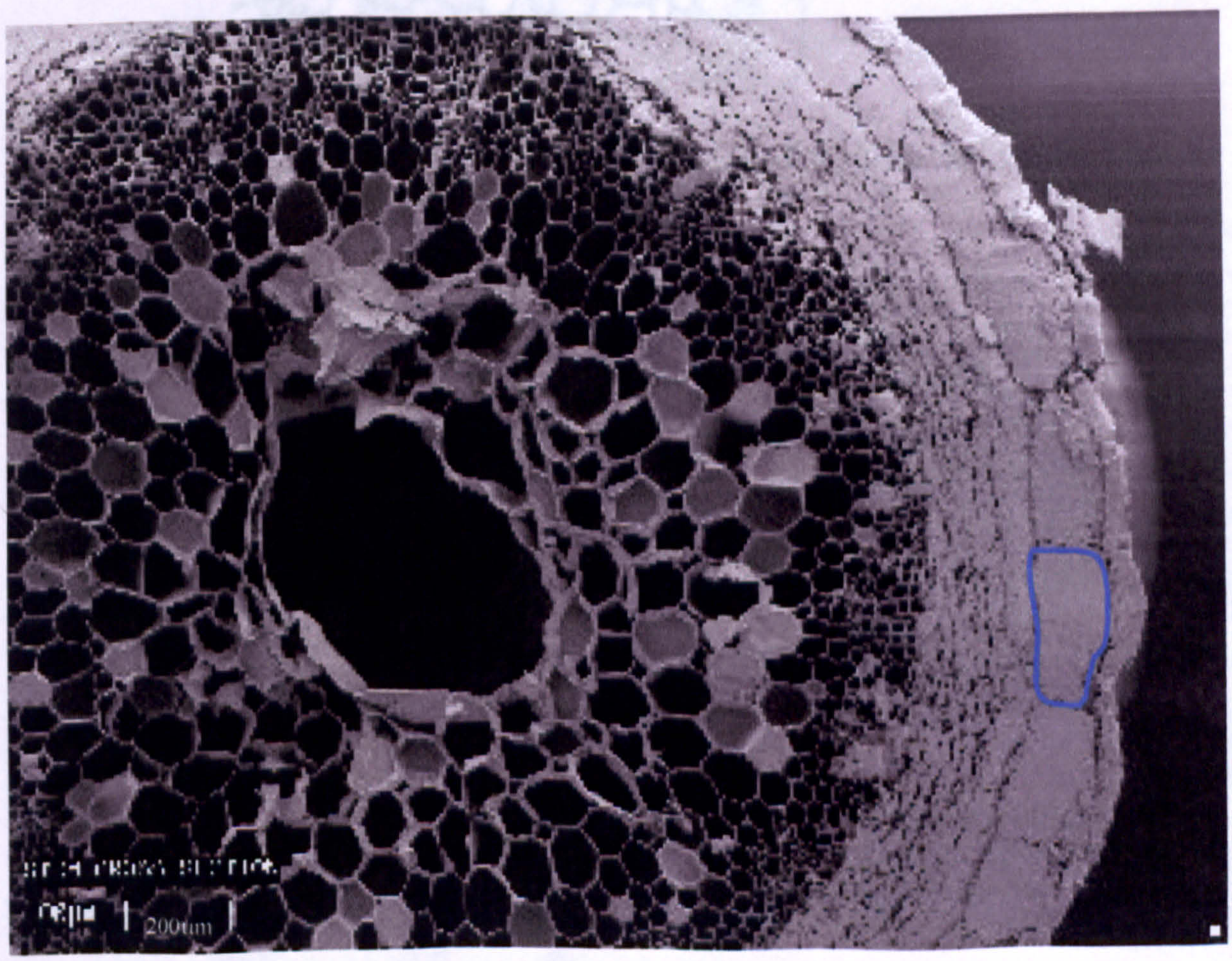


Plate 7. Transverse section of flax stem, light microscopy above and SEM below, (individual fibre bundles outlined in blue).

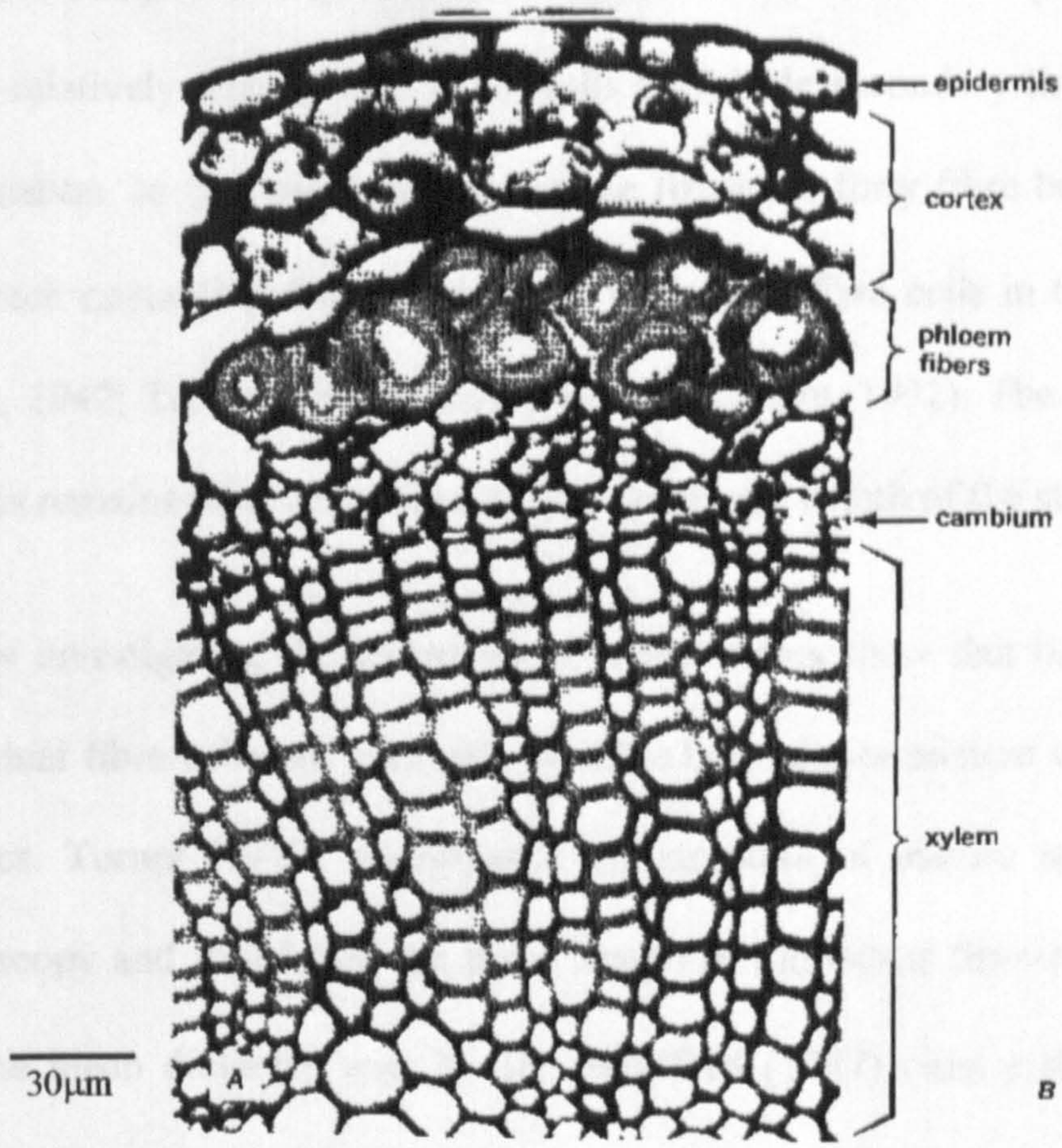


Fig. 1. Diagram showing a transverse section of a typical flax stem

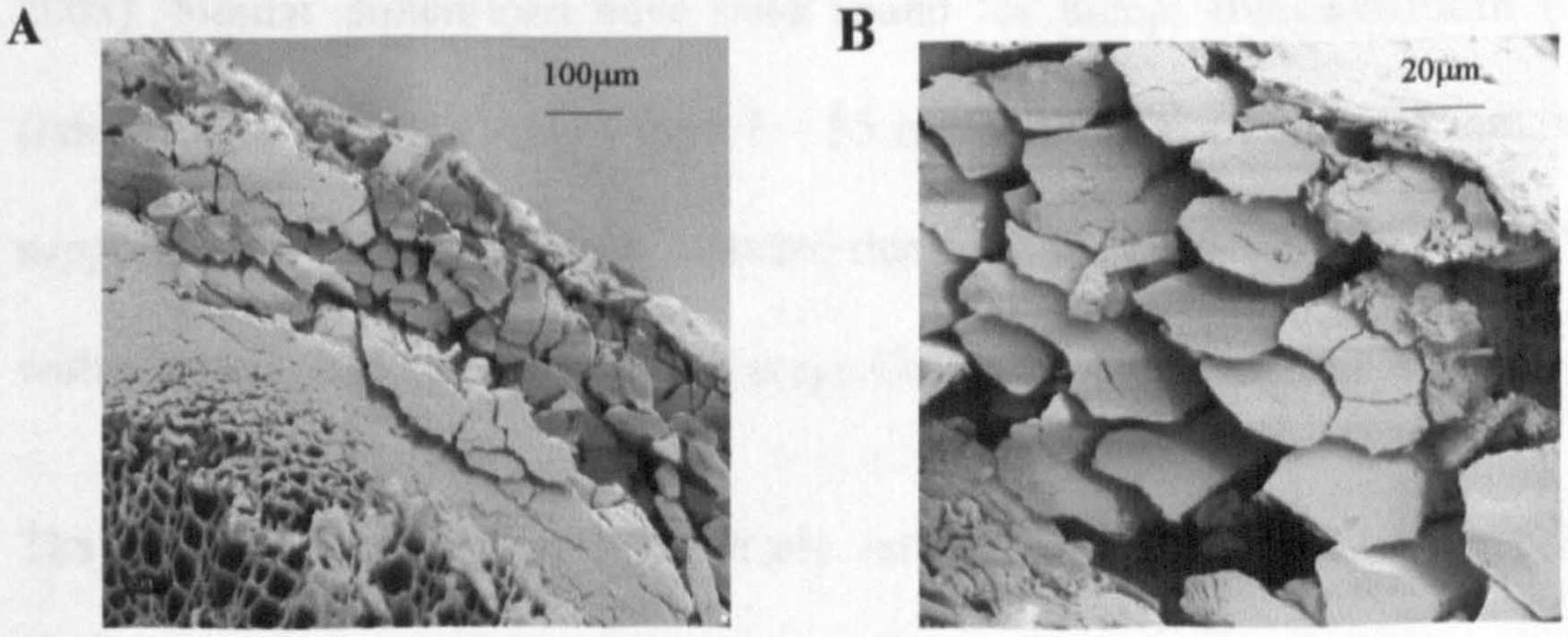


Plate 8. SEMs showing dissociation of fibre bundles in retted flax stem (A), and separation of ultimate fibre cells (B) with debris attached to cells close to the outer edge of the bundles, but less attached to fibre cells originating from within the bundles.

(parenchyma or chlorenchyma), below which the bundles of fibres are located. The fibre bundles are separated from each other by columns of parenchyma cells. These relatively simple cells have walls with little secondary thickening and no lignification. In flax there are on average fifteen to forty fibre bundles per stem, with each containing from ten to forty individual fibre cells in the cross section (Hock, 1942; Turner, 1949; Sharma & Van Sumere, 1992). The number of fibre bundles remains relatively constant throughout the length of the stem.

Studies investigating the morphology of flax stems show that the dimensions of individual fibre cells are generally variable but quite consistent within individual samples. Turner (1949) investigated the structure of mature stems using light microscopy and found that the mean length of individual fibre cells was 27 mm and the mean diameter was 23 μm. Whitford (1947) cites a report by Dodge, stating that mean cell length was 25 mm. More recently, typical average length of individual fibre cells has been reported to average 20 – 50 mm (Morvan *et al.*, 2003). Similar dimensions have been found for hemp; Ilvessalo-Pfaffi (1995) states that fibre length ranges from 5 – 55 mm with an average of 25 mm, which supports Whitford (1947), who reported that fibre length ranged from 5 – 55 mm with an average of 20 mm and an average fibre diameter of 22 μm.

The fibre cells are "cemented" firmly into bundles by carbohydrate polymer substances such as pectin (Talmadge *et al.*, 1973; Morvan *et al.*, 2003). There are many contrasting reports in the literature concerning lignification (the deposition of a mesh of lignin into the cell wall structure) of the fibre and the role it plays in maintaining the integrity of the fibre bundles; some workers have reported an important role (Akin *et al.*, 1997), while a recent review by Morvan *et al.* (2003) found very little evidence of lignin involvement in flax fibre bundle cohesion.

Their conclusion was that a complex pectic matrix possibly involving protein cross-links was responsible for fibre bundle integrity. Similarly, Morrisson, *et al.* (2003) found virtually no evidence of lignin in flax fibres and concluded that the small amounts of lignin in fibres reported in earlier studies could have been due to residual shive (core of the stem) or contaminating remnants of the cuticle. However, in a study of hemp, Wang *et al.* (2003) report a residual level of lignin in fibres after chemical treatment. It has been reported that cells of the cortex may also become impregnated with lignin and other similar aromatic polymers (Morrison *et al.*, 2003) and this may in part explain the adhesion of cell wall debris, which appears to remain attached to the surface of fibres following retting (Plate 9).

The boundary between the phloem and the cortex is not clearly defined in flax and hemp stems, but in some species there is a definite boundary called the endodermis, where cells have specific characteristics, such as high numbers of starch grains or a Casparian Strip. The cells in this specialised layer have walls that may be impregnated with aromatic polymers such as suberin, an aromatic polymer of lower molecular weight than lignin but with similar properties, which regulates the transverse movement of water. Very often the endodermis is not present in the stems of plants, but it is highly developed in the roots of most species and is continuous with that of the stem when it is present. Thus the innermost layer of cells in the stem cortex, which are contiguous with the outer cells of the fibre bundle, occupies the position where the endodermis would be. However, some species develop an endodermis only as the stem matures, or retain a vestigial endodermis throughout their lifetime; the waxy substances associated with the endodermis develop rapidly from flowering onwards. It is likely that flax

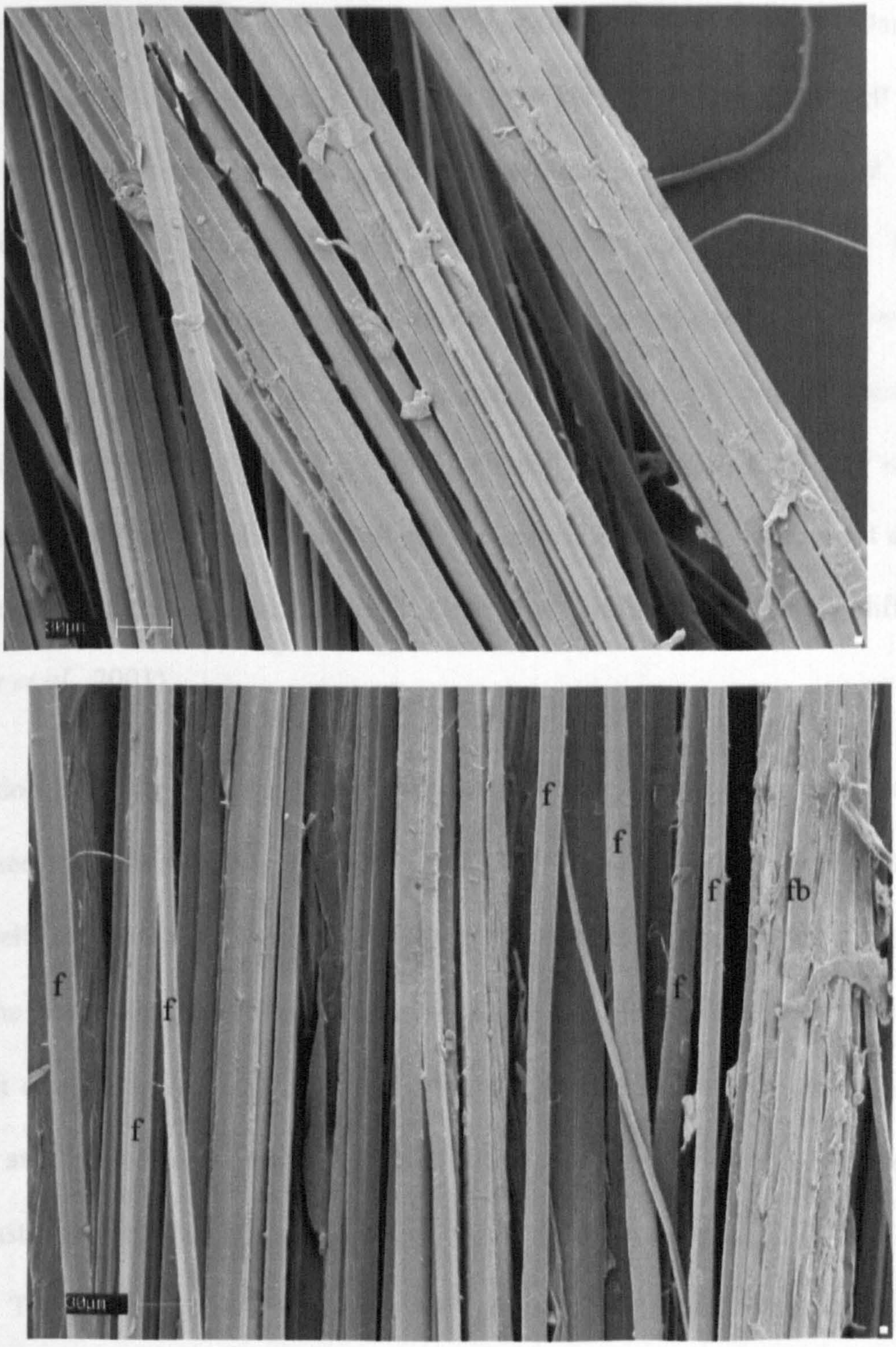


Plate 9. Stages in dissociation of fibre bundles from a flax stem and bundle division to release individual fibre cells. Top shows fibre bundles (fb) mostly intact but dissociated from stem, with attached cell debris; bottom shows the division of fibre bundles to release individual ultimate fibre cells (f).

and hemp have a layer of cells similar to an endodermis (Eames & MacDaniels, 1947; Fahn, 1990) and this may also help to explain the adhesion of cell wall debris from the cortical parenchyma cells on the surface of the fibre cells following retting and their subsequent dissociation from the stem (Plate 8). There is also evidence that the concentration and characteristics of lignin also change as the cells mature, particularly after flowering (Keller *et al*, 2001). Thus the adhesion between fibre cells and their adjacent cells (either other fibre cells within the bundle or the surrounding cortical cells) changes, becoming more robust as the cells mature, causing separation of the fibres to become increasingly difficult (Keller *et al.*, 2001).

Extension growth of fibre cells occurs in two phases; initially elongation is co-ordinated in phase with the surrounding tissue cells (symplastic growth), but the fibre cells are also able to continue extension growth, by apical intrusive growth, after the surrounding cells have ceased to extend. The fibre cells extend vertically in both upward and downward directions; the cell tips extend into inter-cellular spaces and force their way between cells. Fibre extension to 18 mm is possible by symplastic growth, but intrusive growth allows further extension to 75 mm total length. The central section of the fibre cell ceases to extend while both ends of the fibre continue intrusive growth, thus the formation of the secondary cell wall begins at the centre of the cell and extends towards the tips, becoming complete only when the fibre cell tips cease extension growth. Downward intrusive growth is more limited than upward intrusive growth due to the maturity of the cells below, while the cells above are still relatively immature, allowing longer intrusive growth (Esau, 1965). This means that the number of fibre cells in a transverse section of stem may gradually increase over time and fibre diameters in

a cross section will include some apparently finer fibres that in reality are immature fibre tips, reducing the average fibre diameter as measured by image analysis of transverse stem sections.

The inter-cellular cement that binds the fibres in position is made up predominantly of pectin and hemicellulose; the hemicellulose is closely associated with the cellulose of the cell wall. The cellulose provides a framework that is impregnated with non-cellulosic polymers, such as lignin, cross-linked with hemicelluloses. The cellulose molecules are long, linear (unbranched), polysaccharide polymers; hemicellulose molecules are shorter, branched, mixed polysaccharide and polyuronide polymers; and lignin molecules are complex, phenolic polymers. Cellulose is largely crystalline, while hemicellulose and lignin are not. Cellulose is organised into micro-fibrils, and is normally stable, lignin is very stable.

Lignin consists of polymerised phenylpropane units, the three most important starting compounds are coumaryl alcohol, coniferyl alcohol and sinapyl alcohol (Lewis *et al.*, 1996). The lignins of different plant groups differ in the percentages of these starting compounds and in the way they are linked. A very stable, three-dimensional molecular network is formed by irreversible covalent bonding; these bonds limit the stretching of the cell wall (and thus growth of the cell). Thus lignin becomes deposited into the cell wall matrix and chemically linked to the polysaccharides adding rigidity and terminating cell elongation (Esau, 1977). The oldest layers of the cell wall are on the outside (primary cell wall) and additional cellulose layers are deposited inside this (centripetally) to form the secondary cell wall, which itself has several layers with differing composition, but this forms only once cell extension growth has ceased. Pectins, delivered in vesicles

originating from the Golgi apparatus dictyosomes (saucer-like stacks of tubules) form the phragmoplast (a complex organelle comprising microtubules and actin filaments), which forms the cell plate at cell division. Cellulose is deposited on either side of this to form the new primary cell walls of the daughter cells. This composite pectin and cellulose structure is called the middle lamella and it appears in the region where two adjacent primary cell walls meet, but it is predominant at cell corners, where there is an abundance of inter-cellular material. Initially, the middle lamella is mostly made up of pectic material, which forms cross-links with the cellulose of the primary cell walls of adjacent cells. Jarvis (2003) showed that many of these links are mediated by calcium, and possibly by other similar anions, (such as boron, magnesium and copper) (His *et al.*, 2001) and these appear to be concentrated at the points of greatest intercellular stress, confirming a role in adhesion, but removal of these with EDTA (a chelating agent) does not cause separation of cells and so other more complex cross-links are likely to be involved. The middle lamella becomes increasingly impregnated with insoluble calcium pectates (Turner, 1949) and lignified with age (Esau, 1977). Lignification begins in the primary cell wall at the cell corners, but then progresses throughout the primary cell wall and spreads into the secondary cell wall.

Lignification is not well understood, but it has been widely studied in grasses where indications are that ferulic acid acts as an initiation point for the polymerisation of lignin (Iiyama *et al.*, 1990; Hatfield *et al.*, 1999) and that hemicellulose may form a template for both the spacial orientation of cellulose microfibrils and the coupling of the lignin monomers during polymerisation (Houtman & Atalla, 1995), while certain proteins may contribute specificity to the

coupling process (Lewis, 1999). Lignification of the polysaccharide matrix may affect the digestibility of the cell wall by enzymes and thus have significant implications for the process of retting. The mechanism of reduced digestibility is not well understood (Jung *et al.*, 1999); however, Turner (1949) and Jauneau *et al.* (1994) suggested that lignification inhibited the activity of polygalacturonase, one of the most important enzymes in the degradation of pectins. Lignification of the secondary cell wall is slow and lags behind cellulose production and secondary wall deposition (Esau, 1965). As the fibre cells mature, they develop thick primary cell walls consisting of several layers, each with different proportions of cellulose, non-cellulose and water (Esau, 1977), and they become increasingly lignified (Turner, 1949) beginning towards the base of the young plant prior to the onset of flowering and progressing with age (Sharma, 1986a). Indeed, Fraser *et al.* (1982) studied the effects of glyphosate on the maturation of flax stems and showed that in untreated plants, lignification progressed for 3 – 5 weeks after the beginning of flowering. In a study analysing the chemistry and structure of fibre and core tissue of flax, Akin *et al.* (1996) found that retted fibre contained lignified cell corners and middle lamellae in some regions. A further study by Akin *et al.* (1997), investigating the effect of retting enzymes on the structure and composition of flax cell walls, showed that sporadic lignification of the middle lamella between fibre cells affected the integrity of the fibre bundles. Indeed both Turner (1949) and Akin *et al.* (1997) suggested that the lignified middle lamellae could prevent adequate separation of fibre bundles during processing. It was suggested that lignin was preventing degradation of the middle lamella by preventing access of enzymes. Although some fibres were completely separated from the core of the stem and the cortex they were still connected together in bundles. The presence of small amounts of lignin and remnants of the middle

lamella, may help to explain the structural integrity and tenacity of high quality fine linen fibres. Akin *et al.* (1997) showed that extracellular enzymes that degraded the middle lamellae facilitated the separation of bundles into individual fibres, however, the individual fibres may be weakened.

To a great extent, fibre quality may be determined by the actual anatomy of the fibre bundles. Thicker cell walls, for example in more mature cells, may indicate a better quality fibre; on the other hand, high lignin content often leads to a lower quality fibre (Tiver, 1942; Kirby, 1963) and Sharma (1986a) suggests that lignification reduces lustre, silkiness and elasticity of the fibres. It seems likely that these observations may be directly related to the degree of cortical debris attached to the fibres. In a study comparing the composition of different qualities of flax fibres, Van Sumere and Sharma (1991) found that poor quality fibre contained higher levels of lignin, but also that high quality fibre retained small amounts of lignin.

2.3 Retting

Retting is the process that facilitates the release of fibres from the stems and it is the most crucial stage in the successful production of natural fibres for textiles such as linen (Sharma *et al.*, 1992). It is suggested that the degradation of pectin is the key process in retting (Meijer *et al.*, 1995) and that this is dependent on the activity of micro-organisms (Sharma, 1988). Retting may be considered to be similar to rotting and as such must be carefully controlled if the resulting fibres are to be of the highest quality. Biological systems are notoriously difficult to manage and retting is no exception (Sharma *et al.*, 1992; Kessler et.al, 1998). There are many factors that interact to determine the progress of retting, and the process is not yet fully understood. However, for a fibre production system to be

successful, retting must be monitored to ensure that the straw is harvested and processed at the optimum stage. This is particularly true when the retting process may be relatively slow due to particularly dry conditions during retting, or when the pre-harvest, stand retting technique is employed (see below) and the producer is under pressure to harvest the crop (either for reasons of agronomic timeliness or adverse weather conditions). There is no reliable, objective, in-field technique yet available that will support crop management decisions regarding the stage of retting and suitability of the straw for harvest and processing. Studies over recent decades have investigated many aspects of retting and factors affecting its success. However, monitoring the progress of retting and the suitability of straw for harvest and processing is still largely based on traditional, subjective assessments that have not changed for generations.

During retting, the matrix (gum) bonding the fibres in place is digested, releasing the fibres from the inner woody core of the stem. Brown (1984) and Easson & Molloy (1996), both described the retting process as the most important stage in the successful production of flax fibre and as such it has been the subject of much research, (for a review see Sharma *et al.*, 1988). Retting is closely associated with saprophytic micro-organisms (fungi and bacteria), which colonise the stem during the initial stages of normal degradation. In a study investigating the activity of *Epicoccum nigrum*, Brown (1984) showed that the separation of stem tissues was unlikely without the aid of the enzymes produced by such micro-organisms. During retting, the fibre bundles are liberated from the woody core by the action of polysaccharide-degrading enzymes such as pectinases, hemicellulases and cellulases, which are released by the micro-organisms involved. Brown (1984), and Brown & Sharma (1984) both showed that the retting micro-organisms

produced polysaccharide-degrading enzymes and that over-retting could occur when cellulases began to attack the fibres themselves. Brown (1984) showed that in stems artificially infected with *E. nigrum*, pectolytic enzymes reached maximum activity shortly after inoculation and that cellulases reached maximum activity later. Indeed, a high level of cellulase activity indicates a potential for destruction of the fibres themselves (Brown, 1984). Over-retting of the fibre is caused when degradation progresses too far and the individual cells in the fibre bundles become damaged. In a study of the micro-organisms associated with the retting of glyphosate-treated flax, Mercer & Fraser (1986) found that over-retted straw lost its strength and produced fibre that was 15 times weaker than other fibres from normally retted, adjacent areas of the same stem. Specific micro-organisms may be important in over retting and studies by Mercer & Fraser (1986), and Bratt, Mercer & Brown (1988), found that *Botrytis cinerea* was present in over-retted areas of stems. The study by Brown & Sharma (1984) found no evidence of cellulase activity in stems infected with *B. cinerea*, and Bratt *et al.* (1988) suggested that other cellulose-degrading mechanisms, involving hydrogen peroxide release, may sometimes be implicated in over-retting.

Harvesting and retting can be carried out using a number of different techniques. The study by Brown & Sharma (1984) showed that the production of high quality flax was best achieved under anaerobic conditions where extra-cellular pectinases from bacteria retted the stems. Water-retting in tanks, slow-running streams or ponds produced the highest quality fibre and was long considered the best method, but it also caused unacceptable environmental pollution and so has been generally abandoned in favour of cheaper and "cleaner" dew-retting, which employs aerobic filamentous fungi to carry out the degradation of the stem

(Brown, 1984). Other techniques include chemical retting, enzyme retting, and pre-harvest retting where the crop is killed by the application of a desiccant herbicide and retted by the action of aerobic fungi while still standing in the field, prior to cutting. (See Sharma & Van Sumere (1992) for a review).

2.4 Retting systems

Traditionally, the stems were placed in still or slow running water, where the stem tissues would begin to disintegrate as they became colonised and digested by anaerobic bacteria. After a period of time in the water, the stems were removed, dried to curtail retting and ease fibre extraction, and then simply beaten to separate the fibres from the woody core of the stem. The woody core would be smashed to relatively small, light pieces of debris whilst the fibres remained intact in long threads, retaining their strength and integrity. Separation of the fibres from the waste debris was completed by relatively simple secondary processes such as shaking or combing; processes that became highly mechanised over more recent times.

Water retting tends to produce the most successful retting and hence the highest quality fibres, but it does also produce effluent that is unacceptable in modern production systems (Karus & Leson, 1995; Sharma & Faughey, 1999). Thus alternative systems have been considered and widely adopted; dew-retting is the most prevalent system used in modern times. The crop is either pulled (e.g. flax) or cut (e.g. hemp) and the stems laid on the ground in swaths to ret. As the description suggests, the system relies on sufficient moisture in the form of dew from the ground to provide a suitable microclimate for the colonisation of the stems by aerobic micro-organisms, mostly fungi. The suitability of this method is restricted to geographical areas where the climate is conducive to the colonisation

of the stem by appropriate micro-organisms (Easson & Long, 1992). There must be adequate moisture from the dew, coupled with relatively dry weather to prevent the swathed straw becoming saturated by rainfall and rotting in the field before it can be harvested.

The quality of the straw, and ultimately the fibre processed from it, may be variable depending on the micro-climate within the swath and its effect on retting. In many cases the crop is acceptable for processing but does not achieve its highest potential, because the inconsistent quality means that it is graded to the lowest standard of its component grades. The limitations of the dew-retting system led researchers to seek alternative methods of production. One such technique, pre-harvest retting or stand retting, which was developed mainly in Ireland for use in flax, kills the crop by the action of a herbicide and then enables the crop to ret whilst still standing in the field, providing a more robust production system. The moisture content of the standing desiccated straw is less than that of straw laid in a swath on the ground as the standing stems dry out much quicker after rainfall. The microclimate around the stem is also significantly modified as airflow around the stems is increased. Thus, the retting process may be considerably slowed down. A report by Sharma (1986b) showed that stand retting typically took 2 weeks longer than dew-retting. These factors help to ensure that the crop is unlikely to be completely lost to over-retting or rotting before it can be harvested and producers can grow the crop more widely, with greater confidence.

2.5 Pre-harvest Retting

In countries where adverse weather conditions may shorten the harvest window, such as in northern and western Europe, dew retting of flax and hemp has led to reduced yields and lower quality of fibre, and in recent years has often resulted in very high crop losses on a significant scale (Rudkin, 2001; Hobson, 2001). These losses are attributable to either over-retting (degradation of the fibres themselves by the cellulose-degrading enzymes of the retting organisms), particularly in wet seasons, or by a lack of retting in very dry seasons.

In pre-harvest retting, herbicide is applied to kill the plants and encourage the colonisation of the standing, senescing stem tissue by retting micro-organisms, prior to cutting and harvesting the crop (Plates 10 & 11).

2.5.1 Herbicides

Gubbels & Kenaschuk (1981) investigated the effects of the contact acting, bipyridinium herbicide diquat on the retting process. Diquat, conventionally used as a desiccant to aid the harvest of linseed crops, is poorly transported within the plant (translocation) and proved unsuitable for use in flax as it produced uneven desiccation, particularly towards the stem base of the taller flax varieties, where spray deposition was poor. In some cases as little as 20% of the stem was desiccated when using diquat (Gubbels & Kenaschuk, 1981).

In a more recent study, Fraser *et al.*, (1982) investigated the herbicide glyphosate, (N-(phosphonomethyl) glycine), for the desiccation of flax. It was found that plants quickly took up the glyphosate, within hours of application, but its absorption tended to disrupt cell membranes (Harvey *et al.*, 1985). Other workers also showed that cell membranes were damaged by glyphosate and considered

Plate 10. Desiccated flax plots (14 m x 22 m)

Plate 11. Stand-retted flax

that glyphosate could be envisaged as a chemical wounding agent. However, these early herbicide formulations contained cationic surfactants that have since been implicated in the destruction of cell membranes during uptake of the herbicide (Bayliss *et al.*, 1996; Feng *et al.*, 1998). Such effects may aid the penetration of the cortex by fungal hyphae and facilitate the retting process. Indeed, in a study investigating the effects of different retting fungi on flax stem structures, Akin *et al.* (1997) suggested that species of fungi able to disrupt the cuticular surface and thus penetrate the cortex more easily may possess better retting abilities.

2.5.2 Factors affecting the efficacy of glyphosate

Glyphosate is readily transported within the plant as it is highly mobile in the phloem, thus it produces a more uniform senescence and hence a more even retting than the bipyridinium herbicides (Sprankle *et al.*, 1975). Courtney & Robinson, (1982) showed that in glyphosate-desiccated flax, stem moisture content decreased from 70% to 20% within 18 days. However, some studies show that desiccation with glyphosate can also be unreliable. The reasons for this are complex since a number of factors affect the movement of glyphosate within the plant, for example growth rate at application. Sharma (1986b) and Sharma *et al.* (1989) reported that translocation was reduced under dry soil conditions, which led to poor desiccation. Indeed, Harvey & Crothers (1988) investigated the effects of water stress on uptake and efficacy of glyphosate and showed that in well watered plants, moisture content of the stem fell from around 70 % to 40 % within three weeks, but in contrast the desiccation of droughted plants was ineffective. They concluded that soil moisture deficit restricted evapo-transpiration and reduced efficacy of glyphosate by reducing its translocation, rather than its uptake.

The growth stage of the plants at application is also important (eg the maturing seed acts as a strong sink for movement within the plant). Harvey *et al.*, (1985), showed that translocation of glyphosate during seed filling was reduced, leading to poor desiccation of the lower stem. The timing of application is critical for effective translocation and activity of glyphosate. Early applications, before the developing seed becomes a strong sink for assimilates, ensure effective translocation throughout the plant and uniform stem desiccation (Harvey *et al.*, 1985). However, at very early timings the stem may not be fully mature, leading to a reduced yield of straw and fibre. Later applications, during seed development, result in the poor translocation of glyphosate and subsequent variable desiccation of the stem (Harvey *et al.*, 1985). This results in uneven retting and hence unreliable fibre quality.

The optimum timing for the desiccation of flax for long fibre production is thus a compromise between maximising effective translocation of the desiccant herbicide and maximising straw yields. The rate and volume of application appear to be of much less importance, except when the application timing reduces efficacy of the treatment (Harvey *et al.*, 1985). However, Harvey & Crothers (1988) investigated the effects of novel methods of spray application and even where deposition of spray on the lower stem was increased, it did not produce a more effective desiccation, indicating the complexity of the problem.

Courtney & Robinson (1982) suggest that the optimum timing for the application of glyphosate is shortly after the mid-point of flowering. By this stage fibre bundles have formed and fibre cells have begun to mature, but the seed development is in its early stages and does not interfere with the translocation of

the glyphosate. Application at this timing has a profound effect on lignification; Sharma (1986) investigated the effects of glyphosate on lignification of fibres in flax and showed that the process was halted shortly after application. Fraser *et al.* (1982) also found that lignification virtually ceased at the point of treatment. This may have important implications for fibre characteristics.

Glyphosate-desiccated stems are colonised by similar species of fungi to those that colonise dew-retted stems, the sequence of colonisation is also very similar and retting proceeds in a similar way (Fraser *et al.*, 1982; Brown & Sharma, 1984). In a comparative study of the differences between pre-harvest retting and dew-retting, Sharma (1986b) found that pre-harvest retting took 1 – 2 weeks longer than dew retting. This may have been due to the reduced moisture content of the stems and a less humid microclimate around them. Studies by Brown & Sharma (1984), Sharma (1986b) and Bratt *et al.* (1988) have showed glyphosate to possess fungicitoxic properties in vitro. The study by Brown & Sharma (1984) showed that application of glyphosate had limited effects on the colonisation of stems by micro-organisms, with some species (for example *Cladosporium herbarum*) being almost completely unaffected (EC_{50} = 0.31 % v/v). However, other species such as *Phoma spp.* and *Fusarium culmorum* were very sensitive (EC_{50} = 0.015 – 0.033 % v/v). Indeed, a later study by Sharma (1986b) showed a decrease in the growth of aggressive secondary colonisers, such as *Alternaria alternata* and *F. culmorum*, leading to a reduction of over-retting of glyphosate treated stems. Bratt *et al.*, (1988) investigated white, bleached patches, which were associated with localised over-retting on some glyphosate-treated stems, and found that *Botrytis cinerea* was heavily implicated as the main cause. This is supported by evidence to suggest that glyphosate does not inhibit the growth of *B.*

cinerea; in a study investigating the involvement of saprophytic fungi in retting of glyphosate-treated flax, Brown & Sharma (1984) found that *B. cinerea* seemed to be relatively unaffected by the fungictoxic properties of glyphosate, and that, in fact, the fungus rapidly increased after glyphosate treatment.

More recent studies using newly introduced formulations of glyphosate have shown that desiccation can be more effective than with the earlier formulations (Easson & Cooper, 2002). These recent formulations may contain more soluble salts of glyphosate and different adjuvant systems, which allow a more efficient uptake of the active ingredient and its more rapid translocation within the plant (Baylis *et al.*, 1996).

2.6 Decortication

The success of any fibre production system depends on the successful extraction of fibres from the stem (decortication), which in turn is determined by the success of the retting step. If retting is incomplete the fibres will not easily separate from the rest of the stem tissues and the success of subsequent secondary processing will be compromised. The resulting fibres will be contaminated by cell debris, fragmented cell wall material and chemicals of a rather sticky consistency, which affect the performance of the fibre in subsequent, secondary processes. On the other hand, if retting is allowed to continue too far, the stems become colonised by more aggressive micro-organisms that have the ability to digest the cellulose fibres themselves, resulting in fibres which have lost their strength and may be destroyed during the subsequent processing. A proportion of these weakened fibres will be broken during processing (e.g. carding) and result in a higher proportion of fibres that are too short to spin. In either case the resulting fibre will be of reduced value to the manufacturer and customer alike.

The progression of retting is thus crucial in the successful production of natural fibres. Many factors affect retting, including: the species and strain of micro-organism carrying out the degradation (and their ability to produce certain enzymes), the availability of moisture, the level of aeration, the availability of nutrients and minerals, the pH of the system, the ambient temperature and the duration of the retting period. The nature of the crop material itself also has an effect, the maturity of the stem will determine the thickness and the degree of lignification of the fibre cell walls, the constituents of the inter-fibre matrix and the ease of colonisation by micro-organisms.

The identification of the optimum stage of retting for harvesting the stems, and their subsequent processing, has traditionally been determined using subjective assessment by experts, and in many respects this has continued to the present day. These skills tend to be used more to grade harvested straw for marketing purposes, rather than for in-field decision-making prior to harvest. Studies have attempted to identify objective methods for monitoring the progress of retting and identifying more accurately the optimum stage for harvesting and processing of the stems, but no satisfactory technique has yet been reported.

2.7 Previous work

Many investigations have assessed the factors affecting retting, some have considered the implications for decortication, but few have directly measured the ease with which fibres can be separated from the stem of the plant.

The assessment of successful retting has largely been considered in terms of the "quality" of the resulting fibre, often without any definition of the term "quality". In flax, traditional techniques relied on subjective assessments of stem

characteristics (Kirby, 1963). These included assessment of straw maturity, degree of retting, colour, ease of decortication (by hand), divisibility (fineness of the fibre) and handle ("feel" of the resulting fibre) as the main criteria used by grading experts to assess fibre quality, often using a simple rating scale.

In a comparison of subjective and objective methods for assessing fibre quality after dew-retting, Sharma & Faughey (1999) confirmed that subjective methods are still widely used across Europe to define the success of retting and fibre quality. In their study, straw was harvested at the optimum stage for fibre quality and laid out to dew-ret. The optimum stage of growth for fibre quality is very difficult to define, as it will depend on the fibre specification required by the end user and it will be affected by a very wide range of parameters, including agronomic factors such as cultivar, soil fertility, sowing date and plant density, and morphological factors such as plant maturity, degree of lignification, and components of the inter fibre matrix. Sub-samples of straw were assessed to determine the end point of dew-retting using traditional subjective methods. The degree of retting and uniformity of retted areas were assessed visually and manually, and straw samples were given a score between 1 and 9 by the expert grader (1 was regarded as poor and 9 was regarded as optimal). The ease of decortication was similarly assessed using a subjective method to provide a score between 1 and 9 on a similar scale. Although this study found no correlation between the degree of retting and the ease of decortication, it did confirm that the degree of retting (despite its subjective assessment) was an important indicator of eventual fibre characteristics, as it correlated well with a number of the objective assessments, such as certain chemical, physical and derivative thermo-gravimetric analyses. The development of more objective methods for assessing the degree of

retting and the ease of decortication may identify stronger relationships between the two processes and provide very useful techniques to predict the eventual fibre characteristics from a sample of straw.

More objective ways of assessing the degree of retting have been developed in the past. Fried's test (Van Sumere, 1992) relied on a visual score of the fibres released following mechanical agitation in hot water (see Archibald *et al.*, 1998 for details). Seaby & Mercer (1984) developed a hand tool that objectively assessed the degree of retting. This device decorticated stems by pulling them through four closely positioned pins using a hook and spring attachment. The extension of the spring was measured at the point when the stems failed and slid between the pins. The results obtained using this test would be affected by the exact dimensions and positioning of the pins, and the accuracy of the point when measurements were recorded. Another study evaluated spectral methods for objectively monitoring retting in flax using spectroscopic characterisation to investigate internal structure while ignoring colour (Archibald *et al.*, 1998). The two most successful techniques were FT-Raman of the extracted fibres, which measured the decrease in waxes and aromatics and FT-IR reflectance of whole stems, which monitored polysaccharide structures.

2.7.1 Profiled roller decortication tests

Several recent investigations have relied on the mechanical disruption of stems to investigate retting. These studies used equipment based on typical industrial processing equipment, scaled down to laboratory size. The weight of debris removed from a sample of straw after each of several consecutive passes through the equipment is recorded for a large number of passes or until no further debris is removed. These studies do not measure the forces involved, or the amount of

work done, in extracting fibres. The data produced is the average for a number of stems.

The ease of decortication of hemp stems has recently been considered (Keller *et al.*, 2001). In this study green stems, harvested at different growth stages, dried and chemically de-gummed, were decorticated by passing them through a series of pairs of motor driven, profiled rollers. The weight of shive lost at each successive decortication was recorded but the work done in decorticating the stems was **not** measured. A similar method has been used to compare unretted, medium-retted and well-retted flax straw (Kohler & Kessler, 1999).

The activity of different retting fungi has been assessed in flax by monitoring the progress of retting using a mechanical test to evaluate the ease with which the bast was detached from the woody core (Fila, Manici & Caputo, 2001). This study also employed two pairs of hand-driven, profiled rollers to estimate the efficacy of retting by monitoring the ease of decortication during mechanical processing. This was then followed by a secondary cleaning step, which involved scraping the fibre bundles with a wooden blade and finally a hand cleaning step to remove all the remaining cortex and woody debris attached. The progress of retting was also assessed by the chemical analysis of the residual fibre pectins using uronic acid analysis techniques.

In order to assess the retting process and consequently quantify the effects of various factors on fibre extraction, it is first necessary to develop a more reliable, objective technique to monitor the progress of retting. Retting comprises two aspects, the dissociation of the fibre bundles from the core of the stem and the separation of fibre bundles from each other. The former involves a fracture

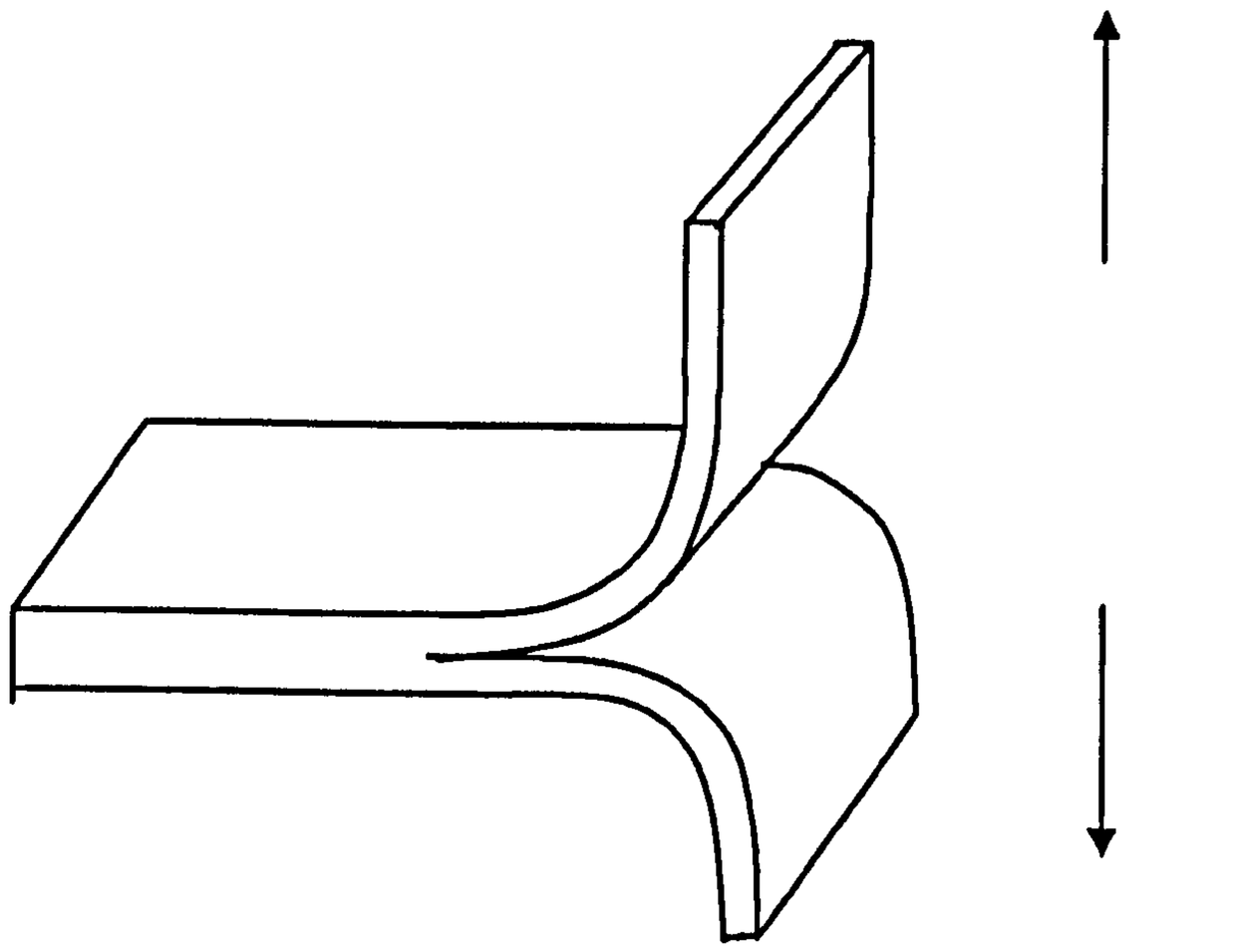
between different tissues and may be the more important step, while the latter may also involve fracture of thin cell walls of the cortical parenchyma cells.

The process by which the fibre bundles fracture from the central core of the stem, may be studied by applying standard engineering principles. Vincent (1990, 1992) suggested a number of techniques to investigate the fracture properties of plants, including peel and "trouser" tear tests. He describes the fracture process, (failure or cracking), in two stages; first the force required to initiate a crack and second the energy required to propagate the crack. Cracks can be propagated in three ways (Fig. 2) mode I: by tension (crack opening), mode II: by shear (in plane slide) and mode III: transverse shear or torsion (out of plane tear). Vincent used both peel tests (mode I) and tear tests (mode III) to investigate the fracture mechanics of grasses and other plants (Vincent, 1990).

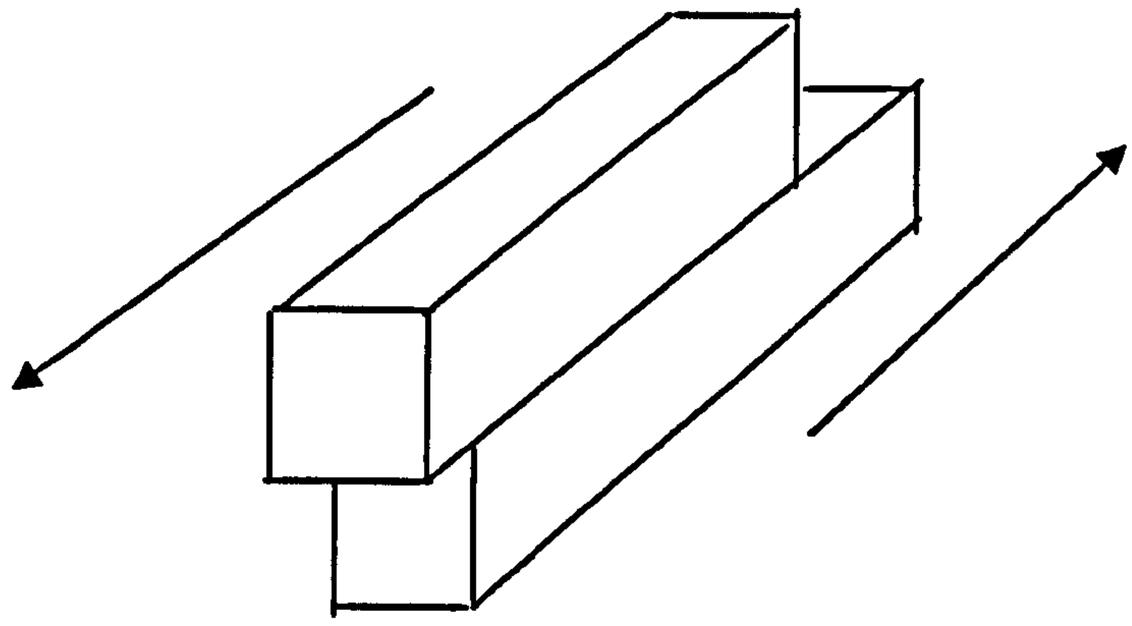
2.7.2 Peel Test

The peel test fracture is a typical tension-propagated crack, i.e. crack opening (Vincent, 1990). Peel tests have been used by engineers for many years to study adhesives, investigating the energy used when layers are peeled apart (see Williams, 1993 for a review). In a study investigating the cell-wall polysaccharides of developing flax plants, Gorshkova *et al.*, (1996) found that it was possible to peel a sheet of fibre and cortical tissue away from the xylem in order to investigate the cell wall chemistry of flax fibres. Peel test methods have been used to monitor the adhesion between different plant tissues, for example Hampshire (1985) studied the feeding of squirrels (peeling the bark from tree twigs) to investigate the work required to separate the sugar-rich outer stem tissues away from the indigestible woody core. However, the peel test method has

Mode I



Mode II



Mode III

Fig. 2. Mode of fracture: tension (mode I), shear (mode II) and torsion (mode III).

not been used to investigate the fracture characteristics involved in the retting of bast fibre crops.

In a peel test investigation of retting in flax, the flexible adherend is the peeled tissue comprising the fibre bundles and the surrounding cortical tissues. It is likely that the fibre bundles in this peel would account for a high percentage of its longitudinal stiffness; the fibres exhibit very high Young's modulus, relating to high stiffness and low extension at failure. It has been suggested that when the flexible adherend is very stiff and the test uses relatively low peel force, the strain energy stored in the peel between the clamp and the peel front can be ignored (Naldrett, 1992).

2.7.3 Tear test

The "trouser tear" test investigates a typical out of plane, shear-propagated crack (Fig. 2); a strip of tissue is torn and the force required to propagate the crack is measured and the work to tear calculated from the area under the force versus crosshead displacement curve (Vincent, 1990 & 1992). The peel section from the peel test has some characteristics in common with the lamina of a monocotyledon (grass) leaf (bundles of fibres run longitudinally, separated by thin-walled parenchyma cells), which exhibit relatively low sheer stiffness (Vincent, 1990). Investigation of this peel strip could indicate the progress of retting by measuring the work required to tear along the peel strip separating the fibre bundles. The relatively simple structure of the peel strip should allow a tear between the fibre bundles to progress with a simple fracture surface, along and between the fibre bundles, thus the tear test can measure the work done when bundles are separated from each other. The tear test measures either the work required to separate (peel apart) the fibre cells from the surrounding tissues, or it measures the work

required to fracture the cell walls of the parenchyma cells between the fibre bundles, or a mixture of both.

2.8. Context

The work reported here formed part of much larger investigations into flax and hemp fibre cultivation and processing, carried out by TEAM Research Group at De Montfort University and funded by EU and UK bodies. In these investigations, I was responsible for the design, planning and implementation of all agronomy work until September 2003. These on-going studies are investigating a wide range of issues related to the production and processing of bast fibre crops, including cultivar, plant density, drilling date, methods of inducing crop senescence (including pulling and herbicide desiccation), harvest timings, methods of decortication, secondary processing and fibre quality characteristics.

In these investigations treatments were randomised in replicated block small plot field trials in crops of hemp and flax, designed and managed to provide straw samples for the investigations. An important aspect of these studies was the development of simple, reliable and reproducible mechanical tests that would enable a large number of samples to be assessed rapidly and objectively.

The profiled roller method of decortication used in several previous investigations was modified and developed to investigate the extraction of fibres from individual flax stems and potentially from hemp stems. An important goal here was to retain the relevance to the practical decortication of commercial crops achieved in previous studies by using the laboratory-scale decorticators, but with modification

of existing techniques to enable more sensitive, accurate and statistically robust assessments to be made.

The suitability of the peel test technique, as used in the investigation by Hampshire (1985) to investigate peeling of bark from twigs, for the investigation of retting in flax was developed further to investigate the work done in peeling fibre bundles from the core of both flax and hemp stems. The peel test enabled the work done in dissociating the fibre bundles from the stem, possibly the most important aspect of retting, to be measured.

The suitability of the trouser tear test was evaluated as a technique to investigate the separation of flax fibre bundles from each other, another important step in fibre production, using a strip of cortical tissue peeled from the core of the stem. This aspect of fibre production is very important in determining the fibre quality at the decortication stage and is related to both the quantity of attached cell wall debris contaminating the fibres and the ease of division of the fibre bundles into finer bundles, or indeed individual fibres. Thus, the tear test data for ease of fibre bundle separation needs to be considered in conjunction with an analysis of fibre quality (fibre fineness and/or amount of debris attached to the fibre). Fibre bundles that are easily separated but retain high levels of attached cell wall debris may be of less interest to the textile end user, but possibly of greater interest to the composites end user (Morrison *et al.*, 2003).

Chapter 3: Materials and Methods

Development of the peel and tear tests for use in monitoring retting

The technique used by Hampshire (1985) was developed further in a study of retting of flax straw that investigated the work done to dissociate fibre bundles from the stems of flax plants (Goodman *et al.*, 2002). This investigation measured the work done in peeling fibre bundles from the stems of two varieties of stand-retted flax over a period of eight weeks following desiccation with glyphosate and then trouser-tear tests were carried out on the peeled tissue.

Commercial plots of Laura and Escalina (Cebeco Seed Innovations Ltd.), five acres of each, were drilled in March at a seed rate of 45 kg ha^{-1}, giving a plant density of around 650 plants m^{-2}. The plots were treated as commercial crops and were desiccated with glyphosate (Zeneca Crop Protection Ltd.) around the middle of August, when seeds were developing within the seed capsules. The glyphosate was applied at a rate of 3 l ha^{-1} in a total spray volume of 200 l ha^{-1} using a commercial tractor mounted hydraulic sprayer with a 21 m spray-boom. Samples of 30 stems were collected from each variety immediately prior to the application of glyphosate and then randomly at weekly intervals thereafter.

Subsequent investigations

Further studies to investigate the work involved in decorticating bast fibre crops were carried out on flax and hemp using different mechanical test methods. Peel tests were used to investigate the dissociation of fibre bundles from the core of the stem in both flax and hemp, and inclined plane decortication tests were also used in flax. The trouser-tear test was used to investigate the separation of fibre bundles from each other in flax.

Hemp

The hemp plots were grown specifically as a field trial with fully randomised plots in a replicated block design at the University farm. A licence was required to grow hemp and the study had to be justified to Home Office officials. Certain conditions of the research licence were restrictive; the trial site itself had to be located in a position well away from general public access and samples had to be partly prepared in the field - all foliage had to be removed from stems and destroyed on site before transport of the stems to the laboratory for analysis. The laboratory site of analysis also had to be licensed and the details of the trial site's location were known to a minimum number of individuals. The permission to grow hemp at the University farm was finally granted late in the growing season, which caused some problems in establishing field trial plots.

An area of land was marked out and cultivated using commercial-scale equipment ready to sow the hemp seed, which had arrived shortly after the license was granted. Conditions were quite dry and so to minimise soil moisture losses, land was worked and plots sowed in a single day. Individual plots were marked out and sowed by hand immediately after soil preparation. The appropriate weight of seed for a single plot was weighed out and spread as evenly as possible over the plot; each plot was sowed individually. Once the seed had been spread, the entire trials area was harrowed to incorporate the seed into the soil to a depth of around 2 cm and rolled down to preserve soil moisture.

Flax

The flax crops were grown under commercial conditions, using commercial varieties, on farms in the east midlands. Sample stems were provided from randomised and replicated trial plots within the commercial crop. Once the crop

began to emerge, a uniform area of the field was selected for the trial and the individual plots were marked out in the existing crop. The plots were treated in the same way as the commercial crop, except for the desiccant herbicide applications. Thus, the plots and blocks were of a size and orientation that fitted with the spray boom-width of the commercial equipment available on each farm.

Previous studies identified glyphosate as the most suitable desiccant for the stand retting technique and so plots were desiccated using glyphosate herbicides, following manufacturers' recommendations, and retted as standing crops (Plates 10 and 11). The original glyphosate formulations were superseded first by Touchdown (Zeneca Ltd.) and subsequently by Quattro (Syngenta Crop Protection Ltd.), these improved formulations offered increasingly more reliable desiccation (Easson & Cooper, 2002; Texflax – LINK project). A sample of flax straw from one farm was also enzyme-retted in the laboratory using a commercially available enzyme with high pectinase content (Viscozyme L) in a solution with EDTA, at buffered pH and constant temperature with gentle agitation (see methods 2.3.2b), in accordance with the manufacturers recommendations.

Generally, random samples of 20 to 30 stems for each treatment were collected weekly for testing and the changes in the ease with which the fibre bundles could be dissociated from the stem (decortication) and separated from each other were measured over several weeks.

3.1 Experimental design

3.1.1 Hemp

a) De Montfort University Farm (now the University of Lincoln Farm), Nettleham, Lincoln.

A small plot field trial was carried out on hemp at the De Montfort University Farm, Nettleham, Lincoln. The plots were sown in late June, at a seed rate of 60 kg ha^{-1} aiming to produce a plant density of around 225 plants m^{-2}. Plots were 8 x 12 m and fully randomised in three blocks; the trial was located within a 6 ha field of set-a-side land. The soil type was a sandy clay loam over limestone. Plant establishment was assessed around 50 days after sowing by counting the number of plants per square metre. No pesticide or fertiliser was applied to the plots.

3.1.2 Flax

a) Lincolnshire College of Agriculture and Horticulture Farms Ltd, Caythorpe Heath, Lincolnshire.

A small plot field trial was carried out at Lincolnshire College of Agriculture and Horticulture Farms Ltd, Caythorpe Heath, Lincolnshire. The soil type was a sandy loam over limestone and the previous crop was winter wheat. The fibre flax variety Laura (Cebeco Seed Inovations Ltd.) was drilled at the end of March, at a seed rate of 45 kg ha^{-1}, using a standard 12 cm row width, aiming to produce a plant density of around 700 plants m^{-2}. Individual plots, of 240 m^2 (10 x 24 m) each, were randomised and replicated in three blocks, with 2 m guard strips between each plot. The crop was managed as a commercial flax fibre crop except for the experimental treatments.

<u>Inputs:</u>

Fertiliser: 100 kg ha^{-1} nitrogen shortly after emergence.

100 kg ha^{-1} phosphorus and potassium in seedbed.

Insecticide: 0.25 l ha^{-1} Pyrimet at cotyledons expanded stage and repeated 10 days

later (for flea-beetle control).

Herbicide: 15 g ha^{-1} Ally + 0.5 l ha^{-1} Bromolin when the crop was around 100 mm tall (for broad-leaf weed control).

Plant density was confirmed by counting the number of plants along a 250 mm length of row at ten positions in each of the control plots one month after emergence. The flowering progress of the crop was monitored and the date recorded when the mid-point of flowering (MPF) was reached. The MPF was identified as the stage when the number of unopened buds was equal to the number of flowers + capsules (Harvey *et al.*, 1985) on a random sample of 30 plants. This was not necessarily the chronological mid-point of flowering.

Touchdown (Zeneca Crop Protection Ltd.), the trimesium salt of glyphosate, was applied at 14 days after MPF, at a rate of 3.0 l ha^{-1} in a total spray volume of 200 l ha^{-1} using a commercial tractor-mounted hydraulic sprayer with a 24 m spray-boom.

b) Huit Farm, Earl Shilton, Leicester.

A small plot field trial was carried out at Huit Farm, Earl Shilton, Leicester. The trial

plots (150 m^2 each) were laid down within a commercial crop of flax (cv. Electra), which followed winter wheat. It was drilled towards the end of March at a seed-rate of 45 kg ha^{-1}. The trial area was treated in the same way as the surrounding commercial crop except for the experimental treatments.

<u>Inputs:</u>

Fertiliser: 178 kg ha^{-1} nitrogen shortly after emergence

148 kg ha^{-1} phosphorus and potassium shortly after emergence

Insecticide: 0.25 l ha^{-1} Cypermethrin at cotyledons expanded stage

(for flea-beetle control).

Herbicide: 14 g ha^{-1} Ally + 0.8 l ha^{-1} Bromolin at 100 mm tall

(for broad-leaf weed control)

1.5 l ha^{-1} Laser + 0.25 l ha^{-1} Cropspray at 500 mm tall

(for grass weed control)

The desiccation treatment, Quattro (Syngenta Crop Protection Ltd.) herbicide, was applied at 3.0 l ha^{-1} in a total spray volume of 200 l ha^{-1}, using a commercial hydraulic sprayer with a 21 m spray-boom width. The application timing was aimed at mid-point of flowering (MPF) and was applied on 7th July. In fact it was applied at MPF + 11 days, slightly later than intended due to adverse weather conditions.

Treatments:

1. Untreated Control
2. Quattro (glyphosate) desiccated and stand-retted
3. Pulled and stand-retted

Quattro (Syngenta Crop Protection Ltd), a recently introduced formulation of glyphosate, was selected for the trial as the optimum glyphosate-based desiccant herbicide. In this experiment, stems treated with Quattro were compared with pulled stems. Samples from both treatment were stand-retted, the Quattro-treated stems were stand-retted in *situ* and the pulled stems were stand-retted in a specially constructed wire frame within the untreated plots of the small plot trial area; both were compared with the untreated control stems.

Weekly estimates of stem moisture content determined the rate of stem desiccation following treatment; and the progress of retting was monitored by decorticating individual stems separately on the inclined plane decorticator.

3.2 Mechanical Tests

3.2.1 Peel tests

Standard mechanical peel tests were used to evaluate the work done in separating fibre bundles from the stem in both flax and hemp. In peel tests, the rigid adherend (in this case the woody core of the stem) is firmly fixed in position and the force required to peel the flexible adherend (in this case the fibre bundles and cortical tissues) away from it is measured.

An Instron universal testing machine (model 4443) fitted with a 100 N load-cell was used for all the peel tests. A section of the stem cortex containing several fibre bundles was peeled away from the core of the stem and the peel force recorded. The work done was calculated from the area under the curve of a force versus displacement trace (Fig. 3) and this was referred to as the work to peel. Immediately after the peel tests, thin stem sections through the fracture surface were investigated using light microscopy (Fig. 4). The fracture was also investigated using SEM (Plate12a and b).

Two types of peel test were carried out; hemp stems were clamped horizontally into the jaws of the testing machine and a standard 90° peel test conducted (BS EN 28510-1:1993) (Plate 13), while flax stems were clamped almost vertically into the jaws and a modified 180° peel test carried out (BS EN 28510-2:1993) (Plate 14). Hemp stems are much more robust than flax stems and could be clamped horizontally in the jaws of the testing machine without physical

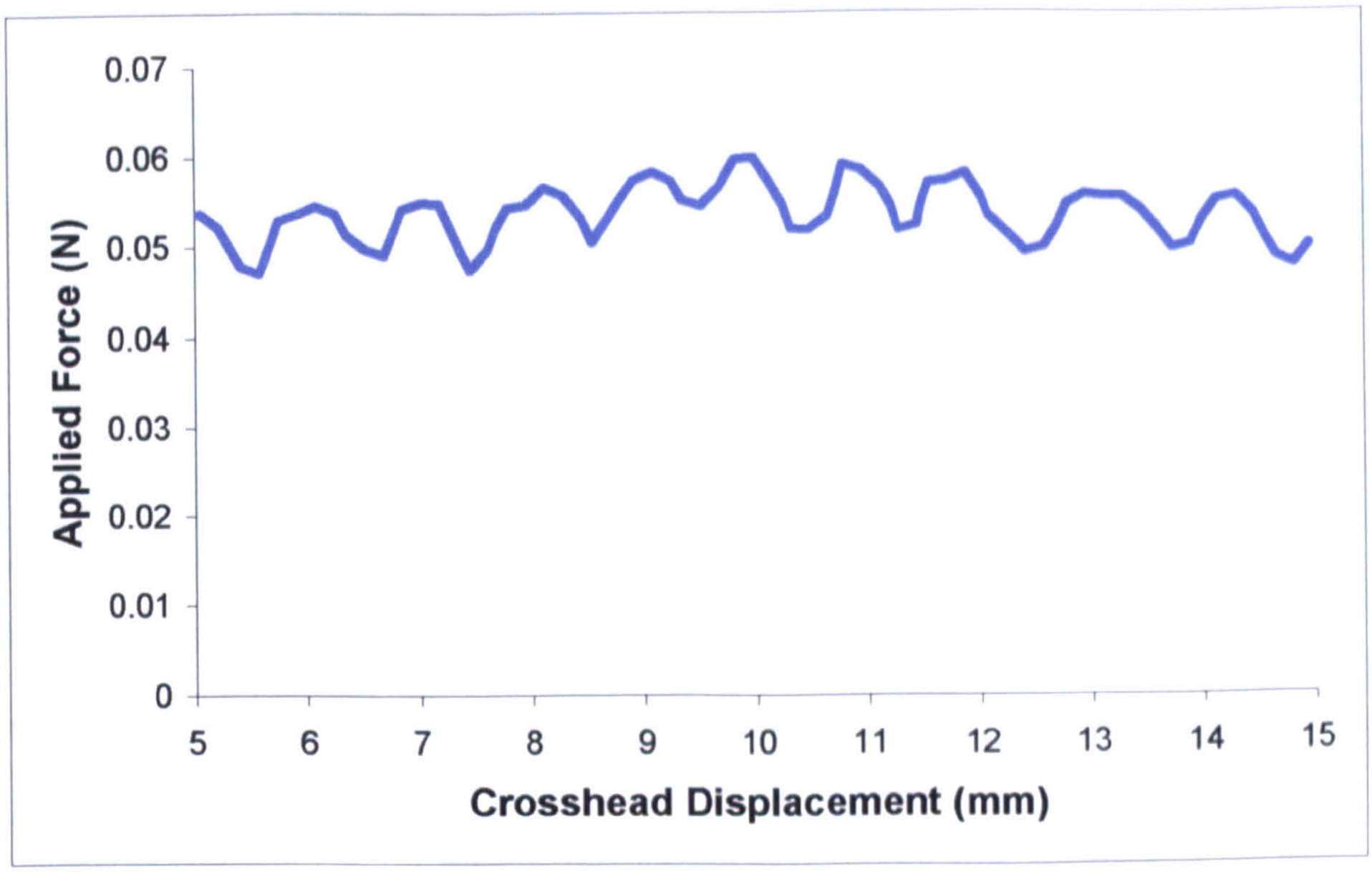


Fig. 3. A typical peel test force versus crosshead displacement trace.

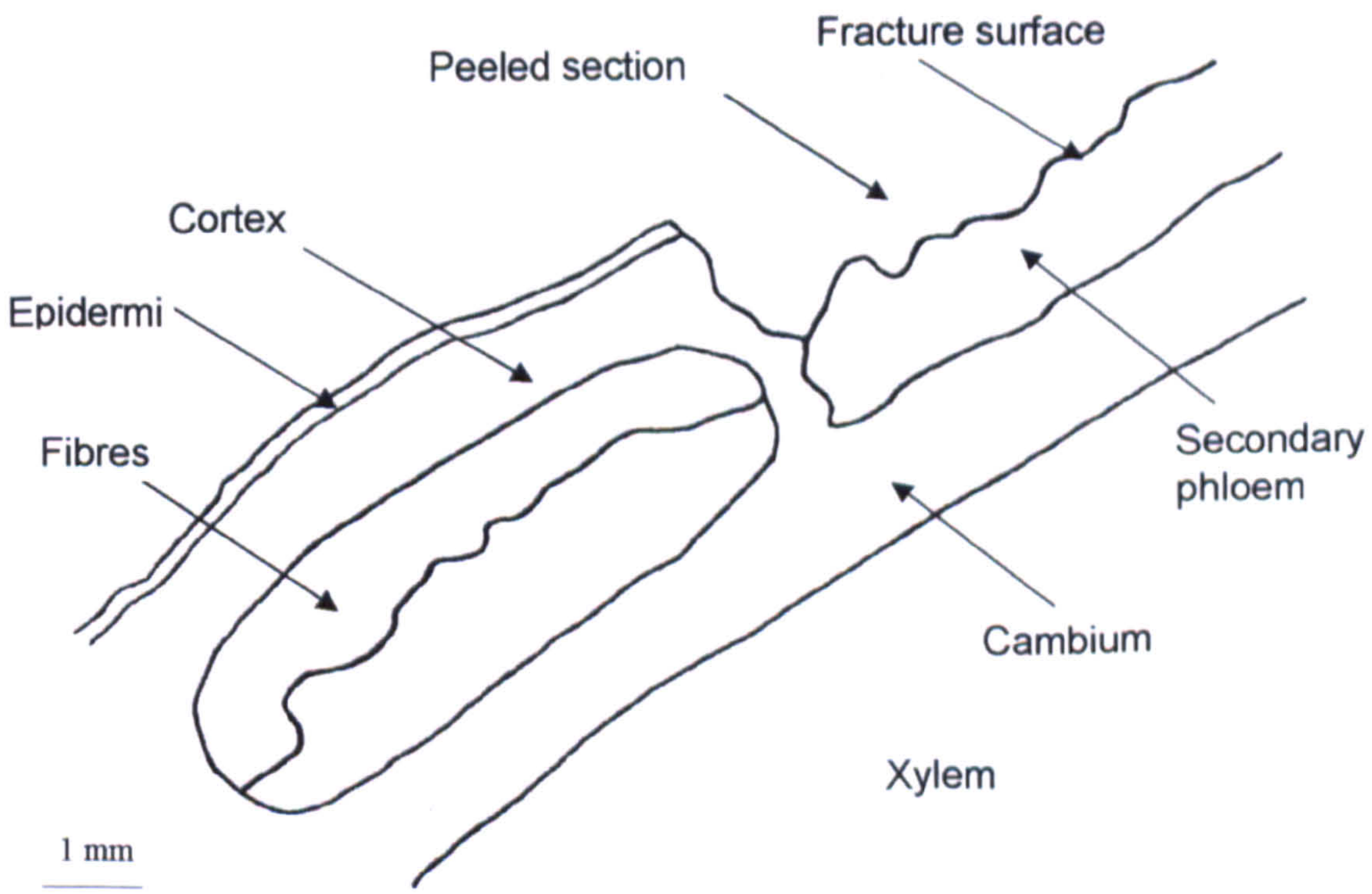


Fig. 4. Diagram of a transverse section through the fracture surface of a peeled hemp stem immediately following the peel test, showing the fracture between primary phloem fibre cells and secondary phloem. Undulations of the fracture surface are clearly visible.

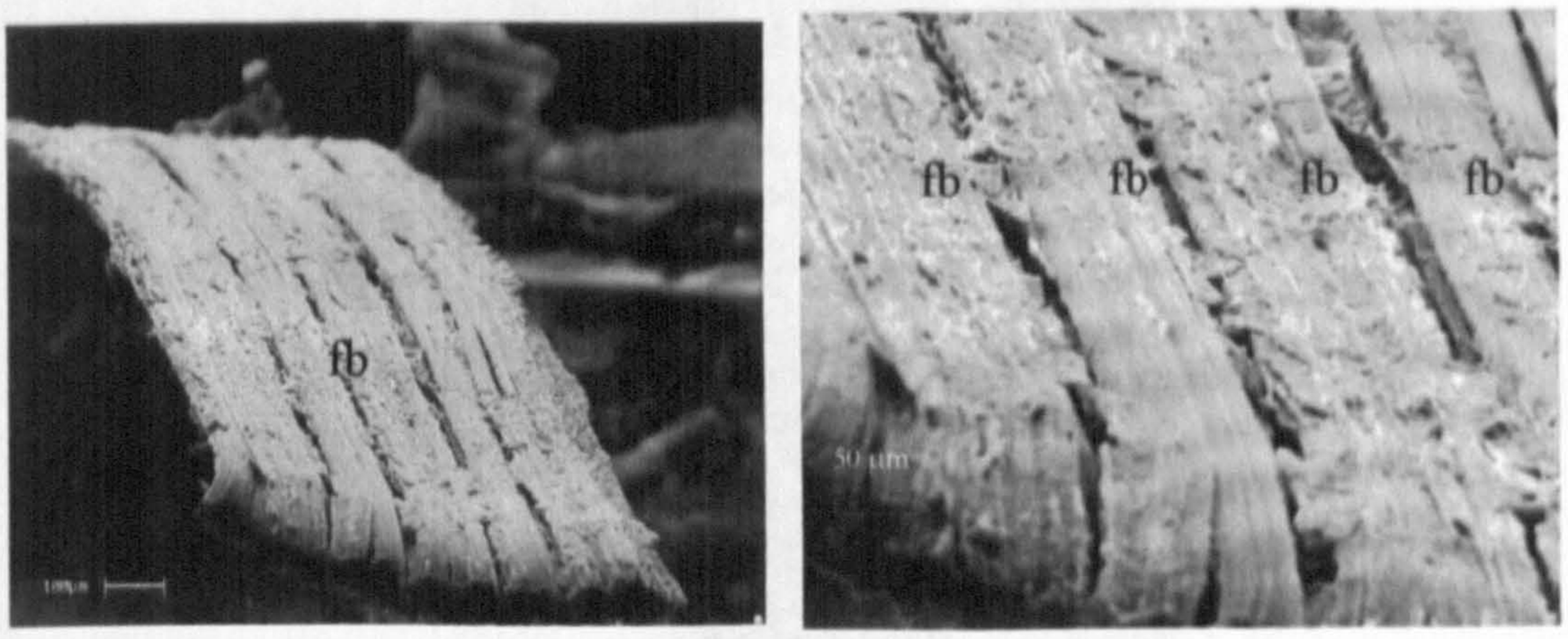


Plate 12a. SEM of underside of peeled flax section; fibre bundles (fb) have been peeled away from core of stem, with debris attached.

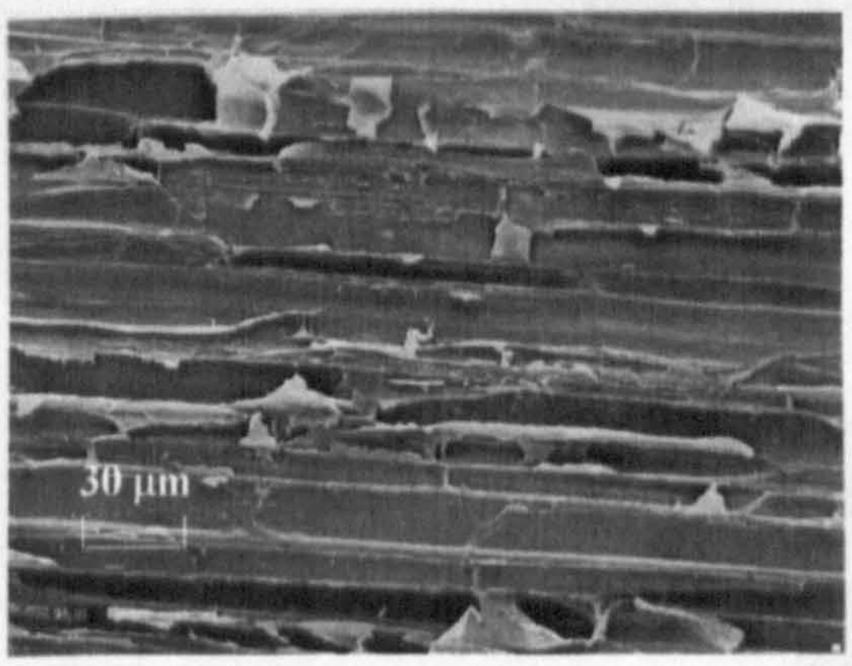


Plate 12b. Surface of shive after fibres were peeled away

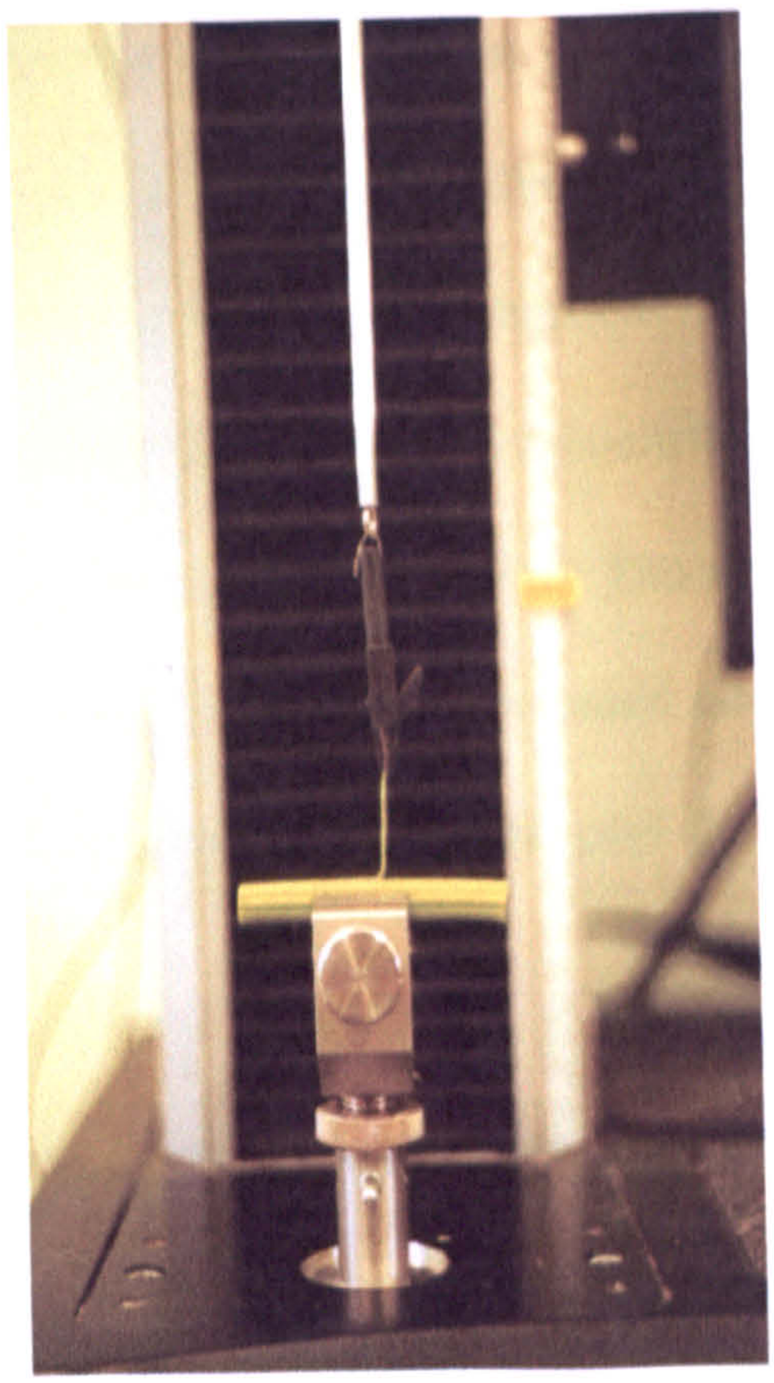


Plate 13. The 90° peel test on hemp

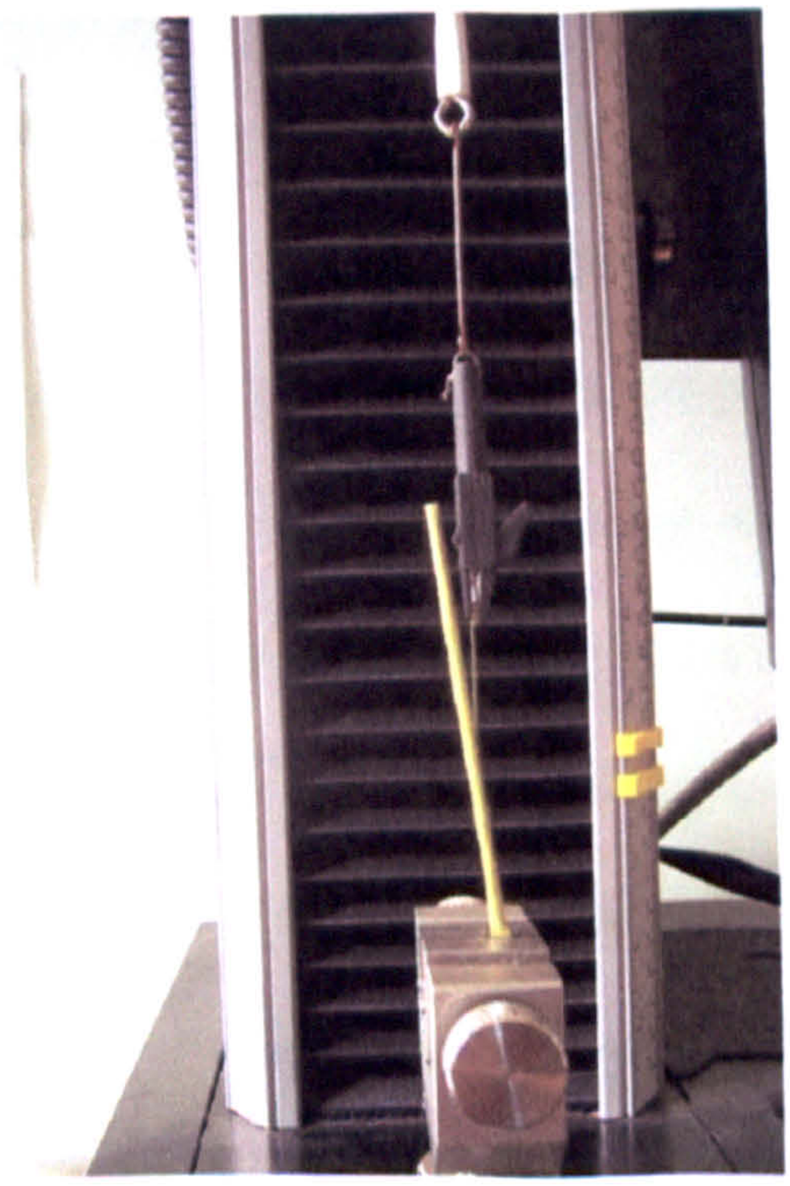


Plate 14. The modified 180° peel test on flax

disruption of the stem tissues within the test area. The relatively delicate flax stems could not be clamped in this way without disrupting the integrity of the stem tissues and compromising the physical characteristics of the stem. Thus they were clamped at one end in a position as close to vertical as possible to reduce the risk of the straw bending when load was applied. The flax samples were clamped as close to vertical as the experimental design would allow without the specimen becoming fouled on the equipment during the peel test (the test geometry was always more than 175°).

The forces involved in peeling the stem were very small in comparison with the Young's modulus of the entire stem, or indeed with that of the peeled tissues, allowing the stored strain energy within either the whole stem or the peel to be ignored.

In both cases an angled incision was made through the outer cortex and fibre bundles using a razor blade and penetrating just to the xylem of the central core. The blade was then lifted, separating the entire peel completely from the tissues below. Thus, a fracture was initiated between the phloem fibre bundles and the underlying tissues; the width of the peeled section was measured using dial callipers. The stem was clamped into the lower jaws of the testing machine and the 20 mm length of lifted peel was attached to the crosshead jaws using a crocodile clip. The tissue was peeled upwards at a speed of 40 mm min^{-1}, an interfaced computer recorded the force required to peel the stem and generated a force versus displacement trace (Fig. 3). The work to peel was calculated from the area under the curve over a displacement of 10 mm, between 5 and 15 mm into the test and was expressed as work done per unit area of the peel exposed (J m^{-2}) (Vincent, 1990). The calculation commenced after a displacement of 5 mm,

ensuring that the force versus displacement trace had stabilised before the calculation commenced. The relatively short displacement allowed multiple tests to be done within the same internode on hemp stems. This method was employed when a series of tests was required, as in the investigations into the effects of peeling angle and stem dehydration.

The standard 90° peel tests on hemp were performed on the relatively flattened sides of the stem, avoiding the deeply ridged aspects of hemp stems. Two razor blades were glued together, 2 mm apart with a plastic spacer between them, to form a simple tool that was used to make two longitudinal incisions through the cortex into the xylem. This method ensured that the longitudinal cuts were uniform and parallel along the length of the sample tested. The tissues between these two cuts were used for the peel tests. This ensured that the work done in tearing through the cortical tissues, separating the fibre bundles from each other, was eliminated from the work to peel calculation.

In these tests the peel was attached to the upper jaw of the testing machine via a tubular aluminium extension piece (500 mm long, 6 mm external diameter, weighing 25 g, with universal joints at each end to allow free movement), which was clamped into the crosshead jaws (Figs 5 & 6). The overall length of this extension piece assembly was 650 mm and it ensured that as the peeling action extended along the stem, relatively constant geometry was maintained throughout the test, any variation in the angle of the direction of applied load was less than +/- 1.0° (Appendix 1) and that the length of peel removed for a 10 mm vertical displacement of the crosshead was increased by less than 1% (Appendix 2). The weight of the extension tube assembly may have tended to exert a force on the peel as gravity pulled it back towards the vertical position, but the relatively

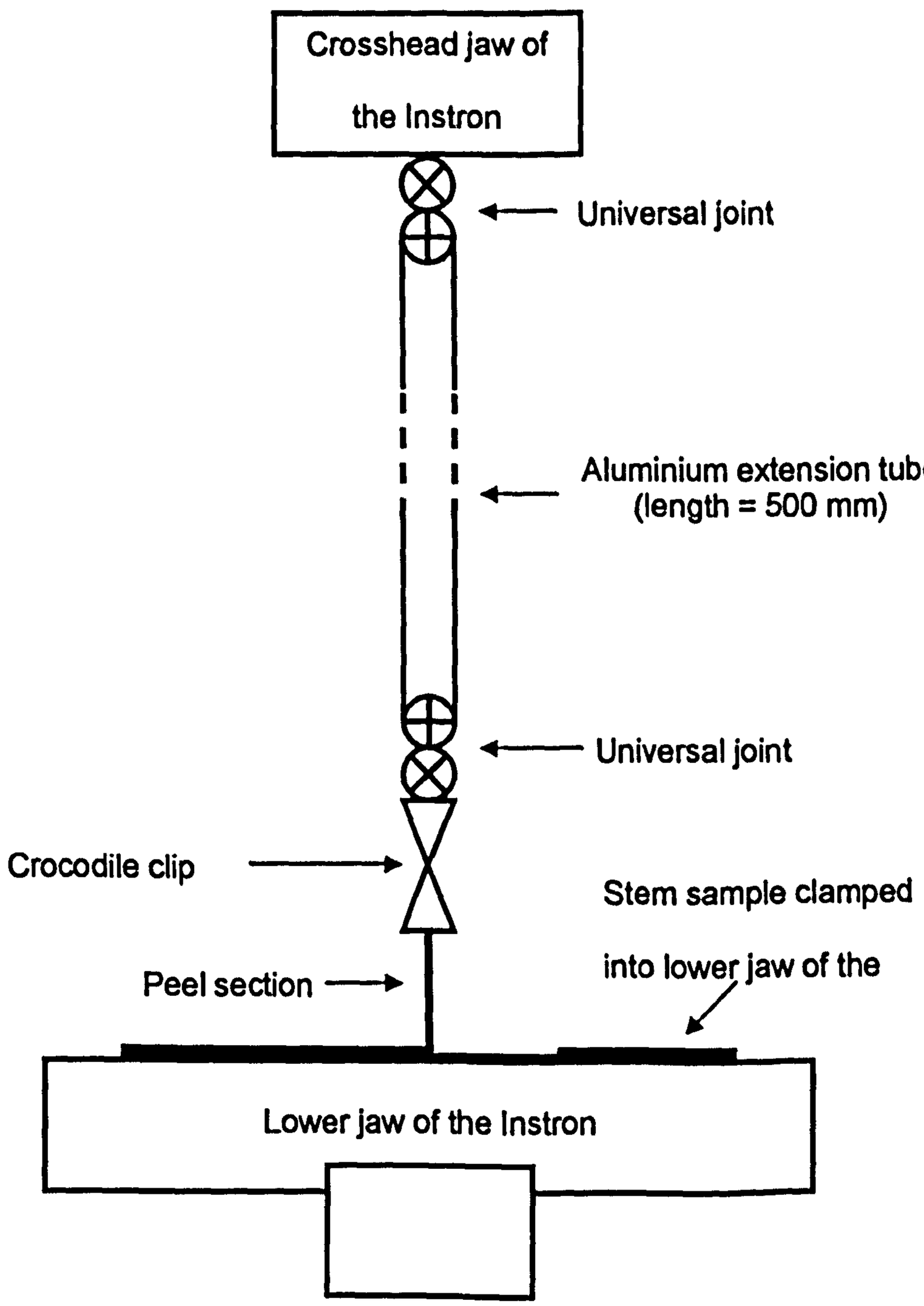


Fig. 5. The test geometry and aluminium extension piece assembly for the 90° peel test.

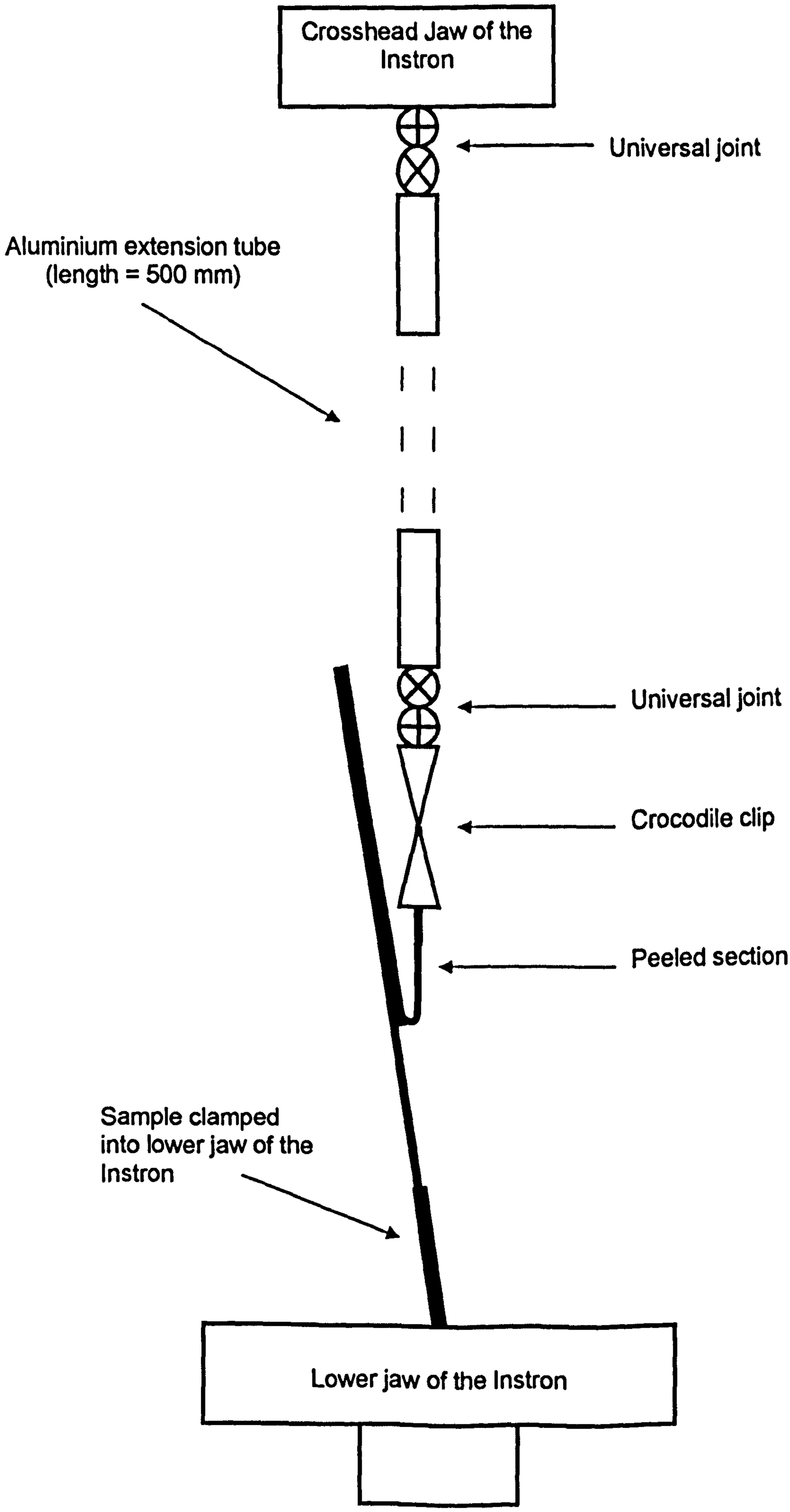


Fig. 6. The test geometry for the modified 180° peel test

constant test geometry would have minimised this error. A crosshead displacement of 10 mm should have produced a peel length of 10.08 mm and this error was also considered to be negligible.

3.2.2 Tear test

Immediately after peeling each strip of tissue away from the xylem, a "trouser" tear test was carried out on the flax peel (Vincent, 1990). A short longitudinal cut around 5 mm long was made in the peeled tissue, (i.e. a crack was initiated), one of the "trouser legs" was clamped into the lower jaws of the testing machine and the other was clamped into the crosshead jaws (Fig. 7). The section of peel was torn longitudinally at a speed of 40 mm min^{-1} using a 10 N load-cell and an interfaced computer recorded the force required to propagate the crack (tearing the peel) and generated a graph of force versus displacement.

Room temperature and humidity were not controlled during the peel and tear tests, but in order to minimise the effects of dehydration on the mechanical properties, stems were stored at 5° C with their bases in water. The sections of stem were peel tested immediately after they were excised from the stem and tear tests were carried out immediately after each peel test.

3.2.3 Inclined plane decortication test

This test method was developed from the profiled roller decortication tests reported in several previous studies (Kohler and Kessler, 1999; Fila *et al.*, 2001; Keller *et al.*,2001). These earlier investigations all used multiple pairs of driven, profiled metal rollers to decorticate bulk samples of straw of known weight (Fig. 8). The samples were passed through a system of rollers several times and the

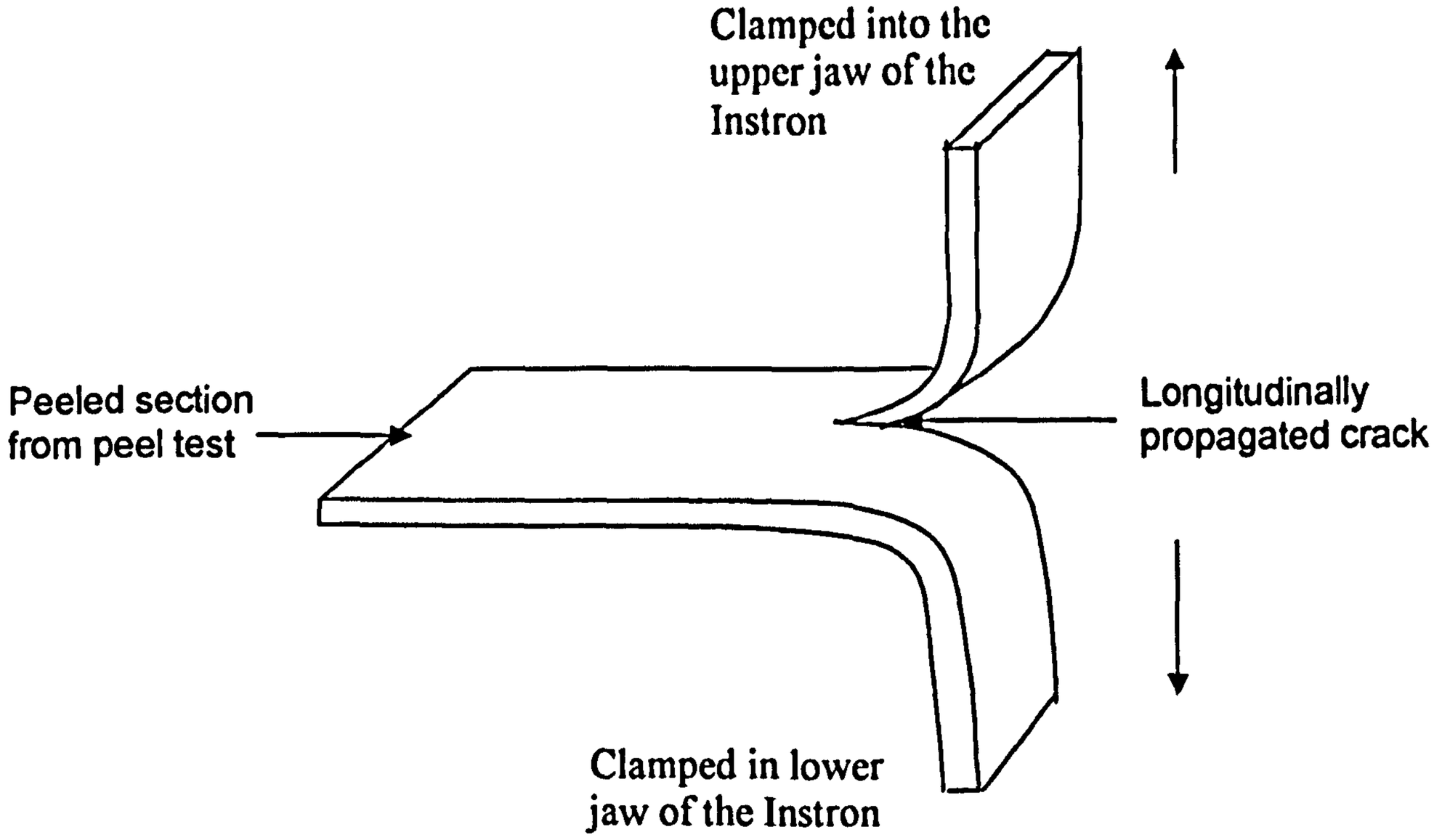


Fig. 7. Geometry of the trouser-tear test.

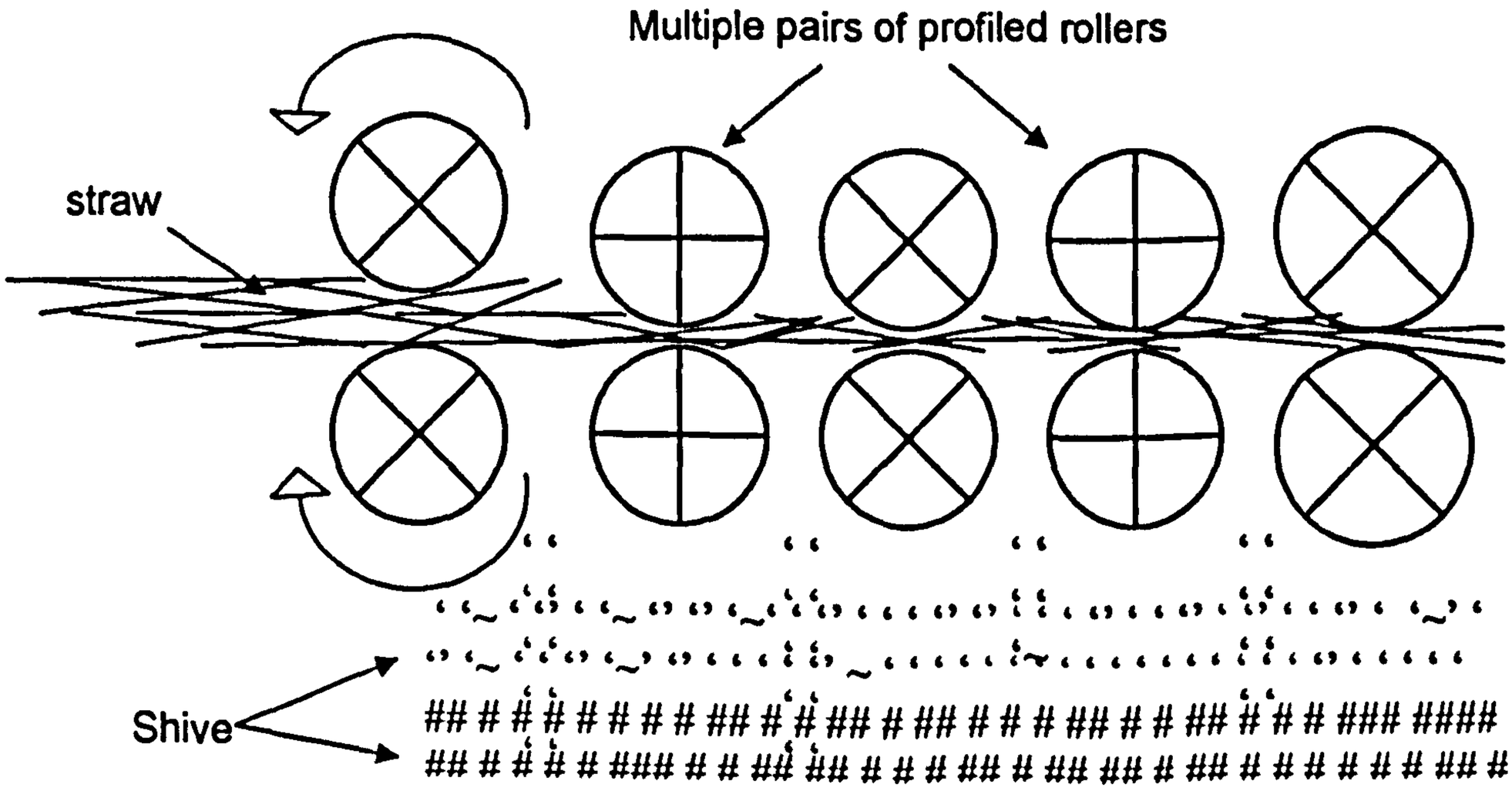


Fig. 8. Schematic diagram of typical roller decortication system with multiple pairs of profiled rollers (not to scale). In laboratory-scale decorticator, rollers would be approximately 10cm diameter.

sample re-weighed after each pass. The woody core of the stem was fractured into small particles and the mixture of fibre and debris was shaken and the non-fibre fraction was allowed to fall away from the fibre sample. The reduction in weight of the retained sample was calculated by subtraction and recorded. A single weight reduction was recorded for one pass through several pairs of rollers, each with its own profile and clearance. The remaining material was assumed to be the total fibre content of the sample and the weight loss was presumed to be non-fibre material of the stem core and outer cortex. However, the driven roller systems may be quite severe and the level of mechanical work applied to each component of the sample is relatively consistent, despite the inconsistent nature of the sample, thus the resulting decortication is at best a compromise. Some fibre may be lost in the debris while some non-fibrous material may be retained in the sample due to the bulk passing through the system. The variability in the process is considerable. Nonetheless, this method does allow the degree of retting for different samples of straw to be compared, but no estimate of the energy required or the work done to decorticate the sample is provided.

In order to improve this method, the basic principle of profiled rollers was retained, but considerably modified. The multiple pairs of rollers were replaced by a single roller, and the mechanical drive was relinquished in favour of a gravity driven system. Instead of two inter-meshing counter-rotating, profiled rollers, the modified system used one roller, a precisely machined timing gear, and its congruously profiled drive belt, 85 mm wide with a pitch of 15 mm and "cog" depth of 6.5 mm (Fenner HTD timing belt, code 2800 14M 85). The drive belt replaced the bottom roller of the pair of rollers and the straw sample was

decorticated between the roller and the drive belt surface. To decorticate the straw sample between the roller and the drive belt; the drive belt was fixed to an inclined plane, which the timing gear was rolled down and over the straw sample (Plate 15). The problem of using bulk straw samples with their innate inconsistency was overcome by decorticating individual stems separately. In order to decorticate the straw effectively, sufficient mechanical work had to be imparted to the sample of individual stems by the roller.

The initial weight of the timing gear was 5 kg and trial decortications showed that this weight was acceptable for the purpose of this investigation. A single pass did not effectively decorticate samples unless they were very well retted, while up to five passes were required for less well retted samples. Neither of these extremes was of importance in this study and typically-retted samples were readily decorticated by 2 or 3 passes through the system. The weight of another timing gear was reduced to 2.85 kg by machining out its centre and further trials carried out with this reduced weight gear showed little difference in efficacy. This lighter gear was used for subsequent investigations, its weight was still much greater than that of the sample being decorticated, (around 30,000 times greater), and the loss of momentum as it rolled over the sample was considered to be negligible.

This modified technique was used to decorticate individual straw samples and, as in previous studies (Kohler & Kessler, 1999; Keller *et al.*, 2001 and Fila *et al.*, 2001), the sample was decorticated using several passes through the system, with the sample weight being recorded before and after each pass.

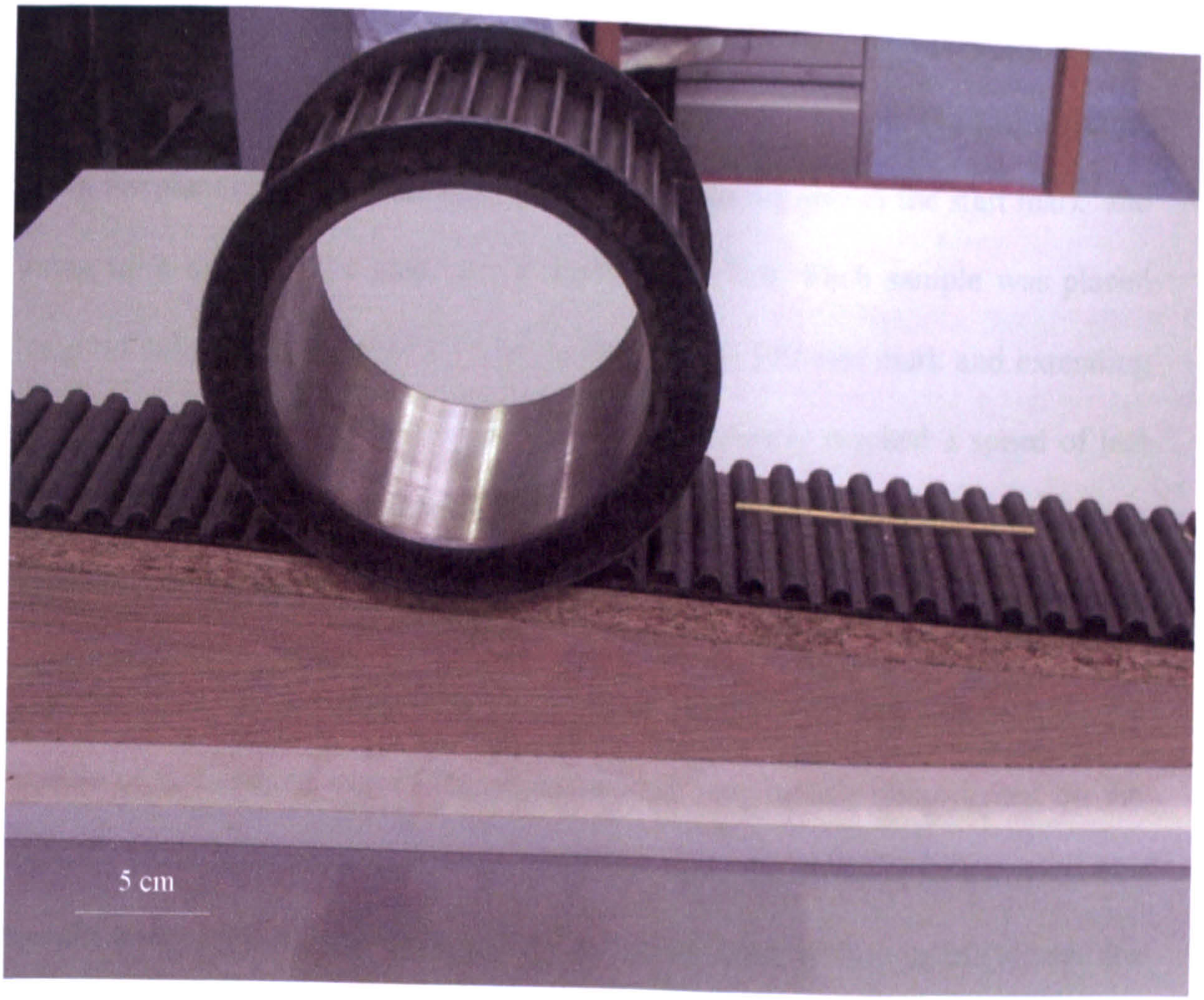


Plate 15. Profiled roller, inclined plane decorticator.

The inclined plane was designed with a 1:10 slope, with a start position marked for the gear and a decortication position marked for the sample (500 mm further down the plane); each decortication pass began with the gear at the start mark and lining up a mark on the gear with a mark on the belt. Each sample was placed longitudinally on the belt with its end level with the 500 mm mark and extending away from the start position. Thus, the gear consistently reached a speed of just under 1 m sec^{-1} before impacting the sample at the start of each decortication pass, imparting reproducible decortication energy to each sample.

Samples of straw for testing were prepared by excising 150 mm lengths from the middle of undamaged stems. The samples were individually decorticated on the inclined plane decorticator. Prior to decortication, the straw samples were air-conditioned at 21° C and 65% relative humidity until stable; initially, samples were weighed on a fine balance every 30 minutes until there was no further change in weight; later samples were conditioned for at least 24 hours. The weight of each sample was determined using a fine balance at the start of the test and again after each decortication pass; the weight reduction was considered to be the weight of shive removed.

Detached but entangled shive debris was removed from the fibre ribbons of each sample using the low velocity airstream from a suction pump. The ribbons of fibre were gripped firmly and held longitudinally in the airflow for three seconds prior to re-weighing. This was repeated for four decortication passes. After each complete series of four decortication passes, any remaining debris was carefully removed by hand prior to a final weighing, thus the total weight of shive removed during decortication was determined, and the proportion removed by each

decortication pass was calculated, indicating the relative ease of decortication for individual straw samples.

The design of the inclined plane decorticator was suitable for the decortication of flax stems but the profile of the gear and the belt was not suitable for the effective decortication of hemp stems because the gear was lifted out of the ridges of the belt and "de-railed" by the more robust hemp stems.

The inclined plane decorticator and the peel test both investigated the dissociation of fibre bundles from the core of the stem and produced similar ribbons of peel, containing fibre bundles. The inclined plane decorticator also removed some of the cortical tissues, especially on well-retted samples, while the peel test had a negligible effect on these outer tissues of the stem. The inclined plane decorticator could be used to produce a value similar to the decortication index quoted by other workers (Kohler & Kessler, 1999) or to calculate the proportion of shive removed by each pass (Fila *et al.*, 2001); however, it still provides no method for measuring the energy used in decortication. Nevertheless, this method is a step forward from the standard methods used previously and could be further developed to indicate the work done when the fibre bundles are dissociated from the core of the stem.

The peel test measures the work done in peeling the fibre bundles away from the core of the stem and although it does not measure the work done in separating fibre bundles from each other, it provides a useful indication of the work done during decortication.

3.3. Mechanical Investigations

3.3.1 Hemp Investigations

3.3.1 a) The effect of peeling angle on work to peel in hemp - De Montfort University Farm, Nettleham, Lincoln.

Aim: To investigate the effect of the angle of peeling on the work to peel, comparing the standard peel tests at 90° and 180°.

Samples

Samples from each plot were harvested by hand at the start of flowering towards the end of August. Random samples of stems (cv. Kompolti) were pulled from each plot (seven from each block providing 21 in total) and these provided the stems for the peel tests carried out at different peeling angles. Moisture content of the stem was assessed at its mid-point by weighing fresh stem segments (50 mm long) and then re-weighing them after oven drying at 70° C for 5 days.

Peel tests

In order to evaluate the effect of changing the angle between the stem sample and the direction of applied load on the work to peel, the stem samples (150 mm long) were clamped into the lower jaws of the Instron at various angles; the load was always applied vertically.

Separate peel tests were carried out, using the same peel section, on the same stem sample. The samples were clamped horizontally (90° peel test), and the sample rotated to 105, 120, 135, 150, 165 and 175° (as close to vertical as the experimental design would allow; modified 180° peel test). Thus, the angle of the peel test was increased in steps, from 90 to 175° (Fig. 9).

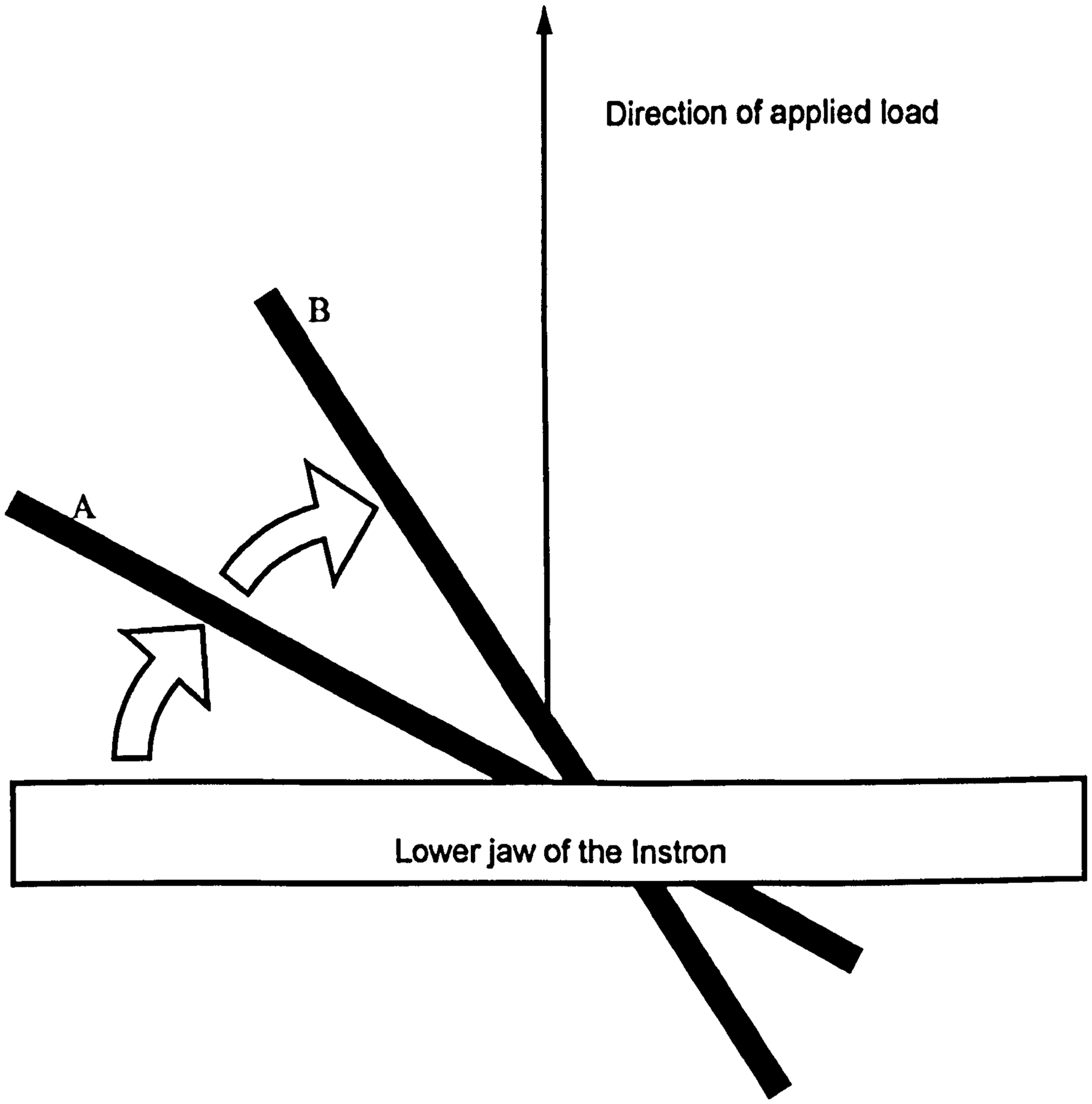


Fig. 9. Peeling angle geometry.

The stem samples were rotated from horizontal to vertical, modifying the geometry of the peel test; the angle was increased in steps of 15° from 90° to 175°. Position (A) represents a peel test at 120 ° and position (B) represents a peel test at 150°.

The prepared stem was clamped horizontally into the lower jaws of the Instron and the peel (20 mm long) was attached by a crocodile clip to the tubular aluminium extension piece, which in turn was clamped into the crosshead jaws of the Instron. The first peel test was conducted as a standard 90° peel test, then the sample was rotated to an angle of 75° between the sample stem and the vertical peeled fibres (this was checked visually using a protractor). The sample was re-clamped and the peel test repeated. This procedure was repeated for each of the 15° steps down to 5° from vertical and the final test (175 °) was carried out. The test geometry would not allow a standard 180° peel test because the peel strip, crocodile clip or the extension assembly would foul on the sample itself, making the results invalid (Fig. 5).

Statistical analysis

Linear regression analysis was used to investigate the relationship between the work to peel and the peeling angle. Student t-tests were used to test for significant differences in the mechanical measurements between the 90 ° and 180 ° tests. All values quoted are means ± Standard Error (SE) to identify confidence limits. The changing data over the course of the investigation required the use of standard error rather than standard deviation in order to allow comparisons between data sets with different means.

3.3.1 b) The effect of stem moisture content on work to peel in hemp - De Montfort University Farm, Nettleham, Lincoln.

Aim: To investigate the effect of tissue moisture content on the work required to dissociate fibre bundles from the core of the stem, in the absence of retting.

Calibration experiment for the dehydration of hemp stem sections in the oven.

In order to carry out a series of peel tests at a range of moisture contents, it was essential to determine the approximate rate of dehydration for the hemp stem samples that would be used in the peel tests.

Stem samples (cv. Kompolti) similar to those to be used in the experiment were dried in a laboratory oven to determine their rate of dehydration. Short lengths (150 mm) were excised from around the mid-point of stems with typical diameters and dehydrated in the oven at 40° C. The decreasing weights of the stem samples were recorded at a range of time intervals over a period of 24 hours until there was no further change in weight. This indicated the time intervals between peel tests to ensure that an appropriate range of moisture contents was covered by the series of tests.

Samples

A random sample of 21 stems (cv. Kompolti), 7 from each block, was collected from the field trial. Sections of stem 150 mm long were excised from around the mid-point of each sample stem and placed in the drying oven at 40° C to dehydrate. Peel tests were carried out after increasing periods of time in the drying oven.

Peel test

In order to investigate the effects of dehydration on the mechanical properties of the stem, in the absence of fungal colonisation, a series of peel tests was carried out at intervals during drying in the oven. As the samples of stem (150 mm long and 6 – 10 mm diameter) dehydrated in the oven they were removed at pre-determined points in time, weighed and a 90° peel test carried out before returning

them to the oven to continue dehydration. Stems with a greater diameter dehydrated at a different rate to those with a smaller diameter, each sample was weighed and tested separately and the work to peel related to the actual moisture content of each sample. Thus, peel tests were carried out on the same section of stem, using the same peel strip, at a range of moisture contents. Each sample was taken from a single internode around the mid-point of the stem to ensure that each peel test was carried out on tissue of a similar age and developmental stage. At peak growth rates, hemp stems can increase in length by over 50 mm per day and so the stem sections being tested would have been very homogenous in terms of developmental age. The actual moisture content of each sample at each peel test could be calculated from the stem weight after the final peel test. The first peel test was carried out on the fresh green stems, whilst subsequent peel tests were carried out after 1, 2, 3, 5, 17 and 42 hours in a drying oven at 40° C; during this time the moisture content of the stem decreased from around 80% to 0%. The same strip of peel was continued for each of the successive peel tests, with a 20 mm section of peel clamped into the crocodile clip attached to the crosshead jaw of the Instron and the excess peel removed each time, but retained for weighing. The final weighing, after 42 hours in the oven, was used to determine the actual moisture contents at which each of the peel tests had been carried out.

Statistical analysis

The results of the dehydration experiment were plotted and the main phases in the data identified visually; Student t-tests were then used to compare the work to peel between the phases. All values quoted are means $\pm$ SE to identify the confidence limits as discussed above.

3.3.1 c) The progress of retting in pulled and dew-retted hemp - De Montfort University Farm, Nettleham, Lincoln.

Aim: To monitor the changes in ease of decortication of dew-retted hemp stems, during the retting period.

Samples

Samples of hemp were collected from the small plot field trial carried out at De Montfort University Farm (now the University of Lincoln Farm), Nettleham, Lincoln and a series of 90 ° peel tests was carried out over a period of 5 weeks, following increasing periods of exposure to dew-retting. Samples from each plot were harvested by hand at the start of flowering towards the end of August, around 100 stems were pulled at random from each plot of Kompolti and Tiborszallasi. These stems provided the resource for the investigation of the change in work to peel during the retting period. The length and diameter of each stem were measured using a metal rule and dial callipers respectively. The stem length was measured from soil level to the tip of the stem and the stem diameter was measured at the mid-point of the stem. The stems tended to have an irregular cross-sectional shape, thus in order to ensure reliable and comparable data, it was important to ensure that the stem diameter was always measured in the same way. Since the smallest diameter tended to be across the flattened sides of the stem this was easier and quicker to measure accurately and this was the measurement recorded.

The harvested stems were randomly laid out to dew-ret on adjacent bare ground in 2 x 2 m square plots, with 1 m strips between the plots (Plates 16 and 17). Stems were laid out individually in an aligned, single layer and turned weekly in an attempt to maintain even retting. Random samples of 21 stems (7 from each

Plate 16. Dew-retting hemp plots (2 x 2 m approx) at The University of Lincoln site.

Plate 17. Kompolti dew-retting plot (2 x 2 m approx.) at University of Lincoln site.

block, cv.s Kompolti and Tiborszallasi) were collected weekly from these dew-retting plots and their length and diameter were measured using a metal rule and dial callipers respectively. In order to investigate the progress of retting in these stems, prepared stem samples (150 mm long) excised from the mid-point of each stem were clamped into the lower jaws of the Instron and 90° peel tests carried out.

Statistical analysis

The Student t-test was used to test for significant differences in the morphological and mechanical measurements between varieties. All values quoted are means ± SE to identify confidence limits as previously discussed.

3.3.2. Flax investigations

3.3.2 a) The progress of retting in stand-retted flax - Lincolnshire College of Agriculture and Horticulture Farms Ltd, Caythorpe Heath, Lincolnshire.

Aim: To monitor the changes in the ease of decortication of desiccated, stand-retted flax stems during the retting period.

Samples

Immediately prior to application of the desiccant, and at weekly intervals thereafter, 30 plants (10 from each block) were harvested and taken to the laboratory for analysis. The length of the stem was measured from the soil surface to the top of the plant using a metal rule and the stem diameter was measured using dial callipers, at a height of 30 cm above soil level.

Samples of 30 stems were collected weekly at random and the progress of retting was monitored using mechanical peel and trouser tear tests. The progress of stem

desiccation was monitored by estimating the moisture content of each stem sample immediately after the mechanical tests had been carried out. The 50 mm long segments of stem were weighed before and after drying in an oven at 70° C for 5 days and moisture content expressed as a percentage of the fresh weight.

Peel test

Modified 180° peel tests were carried out on the flax stem segments, which were clamped almost vertically into the lower jaws of the tensile testing machine. The peel was clamped to the crosshead jaws using a crocodile clip via a connecting piece of dowel (approximately 250 mm long), with universal joints at either end allowing free movement. This ensured that the peeling angle was as close to 180° as possible (always >175°).

Tear test

The segment of peel removed from the flax stems by the peel test was immediately subjected to a trouser tear test that measured the force required to tear the peel longitudinally.

3.3.2. b) The ease of decortication of enzyme-retted flax following increasing periods of time in a retting solution – laboratory based, TEAM Research Group, De Montfort University, Leicester.

Aim: To monitor the changes in ease of decortication of enzyme-retted flax stems during the retting period.

Samples

A sample of straw, collected from the commercial flax crop at Huit Farm, was retted in the laboratory using an enzyme mixture high in pectinase, simulating retting in the field. The central 150 mm section was excised from each stem and

placed in a retting solution comprising Viscozyme L (0.1 ml g^{-1} straw) and EDTA (5 g l^{-1}), at pH 5 (sodium acetate/acetic acid buffer) in a water bath at 40° C with gentle agitation (0.5 Hz).

After increasing lengths of time in the retting solution, sub-samples of ten straws were removed at random for testing. The first sample was removed after 16 hours and subsequent samples were removed after 20, 24, 40 and 44 hours in the retting solution. The samples were rinsed with distilled water immediately to remove as much enzyme as possible and spread out to dry at ambient temperature.

Inclined plane decortication test

The progress of retting was monitored by decorticating individual stems separately on the inclined plane decorticator. The samples were stabilised and tested in a standard air-conditioned environment at 21° C and 65% relative humidity. In order to reduce the risk of the "end" effects observed in initial exploratory work, such as excess disruption of the fibre bundle integrity due to the agitation in the retting solution or enhanced enzyme penetration of sample ends, the central 100 mm section from each 150 mm sample (n = 15) straw was excised and individually tested. The entire series of tests was conducted with and without the low velocity airflow separation of excess shive and debris from the sample.

3.3.2. c) The ease of decortication of herbicide desiccated, stand-retted flax - Huit Farm, Earl Shilton, Leicester.

Aim: To investigate the changes in ease of decortication of desiccated, stand-retted flax stems during the retting period.

Samples

At application of the desiccant, random samples of around 100 plants were pulled by hand from each untreated control plot. These plants were positioned vertically in the wire frames, where they senesced and retted as standing stems, under similar conditions to the herbicide desiccated stems.

Random samples of 15 plants per treatment were taken at weekly intervals, roots and flowering inflorescences were removed and moisture contents estimated by weighing before and after oven drying at 105° C for 4 hrs.

Inclined plane decortication tests

The progress of retting was monitored by decorticating individual stems separately on the inclined plane decorticator (n = 15). The samples were stabilised and tested in a standard air-conditioned environment at 21° C and 65% relative humidity. The low velocity air stream separation technique was used to ensure complete removal of dissociated shive and debris from the sample.

Chapter 4: Results

Development of the peel and tear tests for use in monitoring retting

Goodman *et al.* (2002) reported two distinct phases in the work required to peel flax stems over the duration of the experiment. Initially, during the first 14 days, measurements of work to peel remained constant, with a mean work to peel of 212 ± 7.9 J m^{-2} ($P > 0.05$); there was then a rapid increase in the work to peel to 539 ± 22.0 J m^{-2} ($P < 0.001$) on day 27, which corresponded with a pronounced drop in the moisture content of the stem, from a moisture content of 60% down to 10% as the plants senesced. Finally, despite relatively constant stem moisture content, there was a more gradual, but significant, decrease in the work to peel to a mean of 297 ± 19.8 J m^{-2} ($P < 0.001$). It was concluded that this was the result of the retting process and indicated that peel tests could be used to measure mechanical changes at the interface between the fibre bundles (primary phloem tissue) and the underlying tissue (secondary phloem). Thus, indirectly, it enabled the progression of retting to be monitored.

In this same report, results from the tear test were more variable, but followed a similar pattern. Goodman *et al.* (2002) describe a significant increase in the work required to tear the peel, but didn't take into account the area of the fracture surface. If their quoted results for "work to tear" are related to a typical peel thickness as measured in other experiments (unpublished), an indication of the true work to tear can be estimated. If a uniform peel thickness of around 1.25 mm is assumed, then an approximate work to tear of around 218 J m^{-2} can be estimated (Appendix 1). It is likely that this is an overestimate since the fracture surface is unlikely to be "flat" and so the area will be greater than that assumed due to undulations. This estimate of work to tear the fresh stem peel is very

similar to the work to peel in the fresh stems, which was 212 J m^{-2}. Unfortunately, the effect over time cannot be evaluated accurately since the peel thickness was not measured and it would almost certainly have changed as the plant senesced and dehydrated. However, if it is assumed that the peel thickness remained constant, the work to tear increased to around 360 J m^{-2} as the stems dehydrated and then decreased to around 208 J m^{-2} after several weeks. These values are less than the values found for the work to peel at the corresponding assessment dates, but values for work to tear would increase if, as is likely, the fracture surface area decreased due to dehydration of the tissues, however, an increase of almost 50% would be required for work to tear to approach the same value as the work to peel. It is perhaps worth noting that the stem moisture content became relatively constant after the initial rapid dehydration of the stems and so the decrease in work to tear towards the later stages of the investigation cannot be explained by changes in the area of the fracture surface due to dehydration.

Subsequent investigations

The investigations carried out by TEAM Research Group cover many aspects of bast fibre production. Many of these require the objective assessment of a large number of samples generated by repeat sampling. In order to facilitate management of all the projects' tasks, it was essential to devise rapid and reliable, reproducible assessment techniques. Much of the work in these projects was aimed at improving the extraction of fibres from the stem to produce uncontaminated and largely undamaged fibres (as defined by the industry standards for cotton fibre) for textile end uses, either as a target in its own right or as the starting point for the evaluation of secondary processing methods, and this was in turn dependent upon effective retting. Thus, it was crucial to be able to

monitor the progression of retting objectively in order to allow treatments to be evaluated. This study reports the development of relatively simple mechanical techniques that proved useful in enabling the rapid, effective and reliable assessment of the large number of samples that were generated.

The results reported here fall into two main categories: those that support the development of the techniques and those that validate and illustrate the capabilities of the techniques. The stem characteristics of flax and hemp affected their suitability for the implementation of the test methods and although all the tests were suitable for use on flax, the inclined plane decortication system proved unsuitable for use on hemp. The techniques developed allowed the analysis of many individual samples, for several treatments, to be completed each day by a single worker and the reproducibility of the test methods allowed multiplication of the procedures.

4.1 Morphological measurements

4.1.1 Hemp

4.1.1 a) De Montfort University Farm, Nettleham, Lincoln.

Crop establishment

Seven varieties of hemp, (cvs Beniko [Institute of Natural Fibres, Poland], Kompolti, Tiborszallasi [GATE Agricultural Research Institute, Hungary], USO 14, USO 31, Zolo 11 and Zolo 15 [Ukrainian Institute of Bast Crops, Ukraine]) were sown, but due to late sowing and subsequent dry weather, the plots were slow to establish and bird damage was severe in places. Only the varieties Kompolti and Tiborszallasi produced acceptable, uniform plant densities and these were used for the subsequent investigations. The plant populations established were around 60 plants m^{-2} for each of these varieties.

Plant height and stem diameter

At harvest, Tiborszallasi was significantly taller than Kompolti ($P < 0.01$, df = 12), with a mean stem length of 1294 ± 27.0 mm for Kompolti and 1480 ± 33.9 mm for Tiborszallasi, but there was no significant difference in stem diameter between the two varieties ($P > 0.05$, df = 12) with a mean stem diameter of 8.9 ± 0.35 mm for Kompolti and 8.8 ± 0.27 mm for Tiborszallasi.

After harvesting the stems and laying them out to dew-ret, stem diameter decreased by around 15 % in both varieties as the stem moisture content decreased. During the first 14 days of dehydration, there was a significant decrease in mean stem diameter from 8.9 ± 0.35 to 7.6 ± 0.21 mm for Kompolti ($P < 0.01$, df = 12) and from 8.8 ± 0.27 to 7.2 ± 0.17 mm for Tiborszallasi ($P < 0.001$, df = 12), but it then remained constant around these values thereafter.

4.1.2 Flax

4.1.2 a) Lincolnshire College of Agriculture and Horticulture Farms Ltd, Caythorpe Heath, Lincolnshire

The plant population established was 737 ± 32 plants m^{-2}, mean crop height was 980 ± 9.4 mm and mean stem diameter was 2.6 ± 0.10 mm. Very warm, dry conditions in late spring caused premature natural senescence in the untreated crop, which senesced almost as quickly as the herbicide-treated crop.

4.1.2 b) Huit Farm, Earl Shilton, Leicester.

The plant population established was 726 ± 24 plants m^{-2}, mean crop height was 847 ± 7.4 mm and mean stem diameter was 2.0 ± 0.06 mm.

4.2 Mechanical investigations

4.2.1 The effect of peeling angle on work to peel in hemp (Appendix 3)

Results for 16 of the 21 stems tested are reported, 5 samples did not produce a full set of results and so were excluded from the analysis. In the rejected samples the peeled section did not maintain an acceptable peel width throughout the series of angle tests. Since a series of seven different peeling angles were carried out on each sample, a total minimum "peelable" length of more than 125 mm was required in order to gain a full set of data for an individual stem. In the five rejected cases, the ribbon of peel did not maintain constant width throughout the series of tests, they became narrower before the data set was complete, perhaps indicating damage to, or "faults" in, the fibre bundles at certain positions allowing bundles to split during peeling. Incomplete sets of data have not been included in the analysis.

The work required to peel stems using the modified 180° peel test (i.e. stems clamped in the almost vertical position) was more than 20% greater than that for the 90° peel test (i.e. stems clamped in the horizontal position). The 90° peel test produced mean work to peel values of 198 $\pm$ 16.0 J m^{-2} while for the 175° peel tests this increased to 241 $\pm$ 15.3 J m^{-2} ($P < 0.05$, df = 12) (Fig. 10a). The effect of peeling angle on work to peel for a typical individual stem is shown in Fig. 10b.

The relationship between peeling angle and the work to peel was analysed in more detail; as the peeling angle increased, the work to peel at first remained relatively constant but then increased. Data partitioning established the angle at which the work to peel began to increase. The experimental data was divided into two groups; Group 1 consisted of the data related to the peel tests carried out at angles

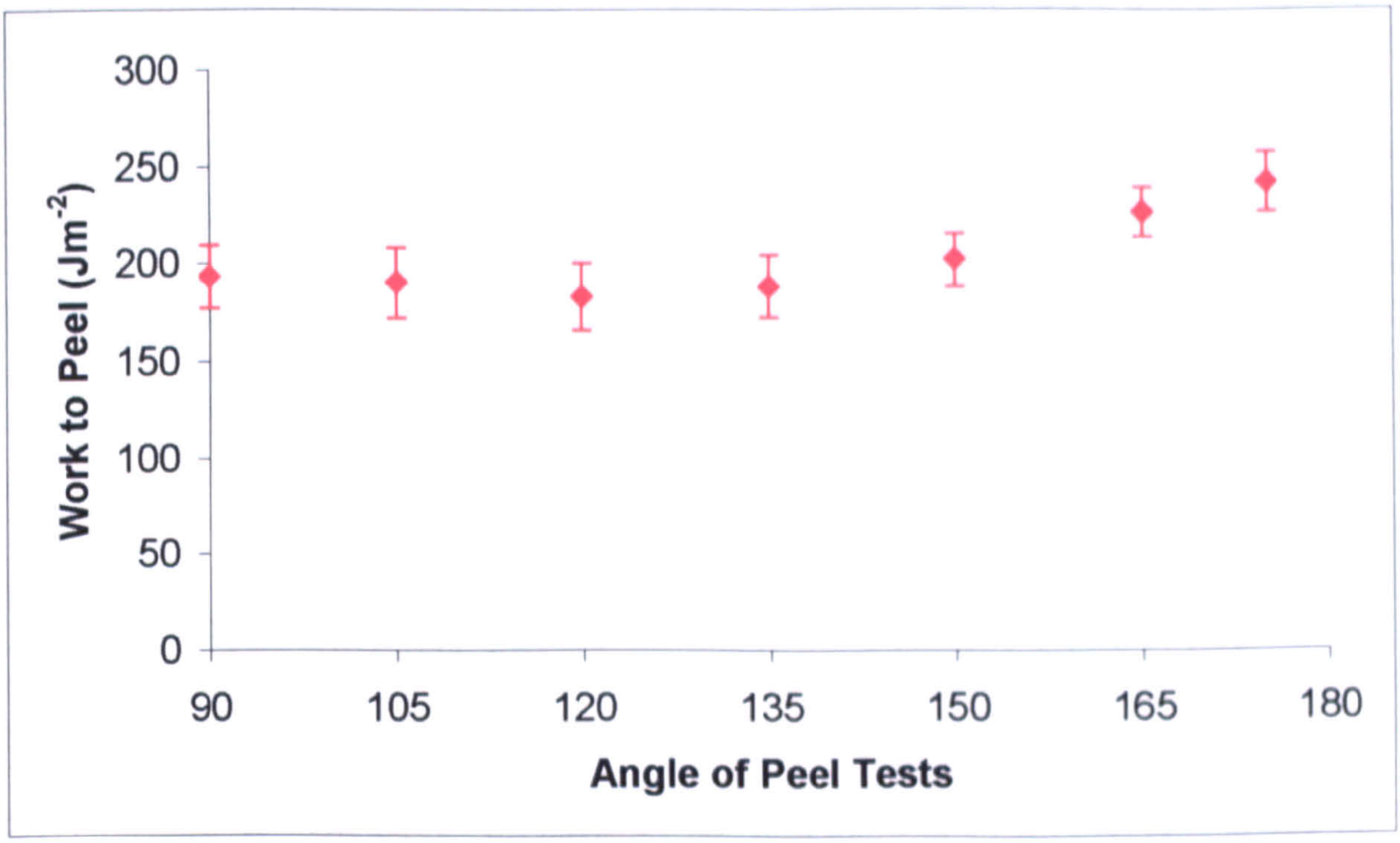


Fig. 10 a. The relationship between the angle of the peel test and work to peel in Hemp.

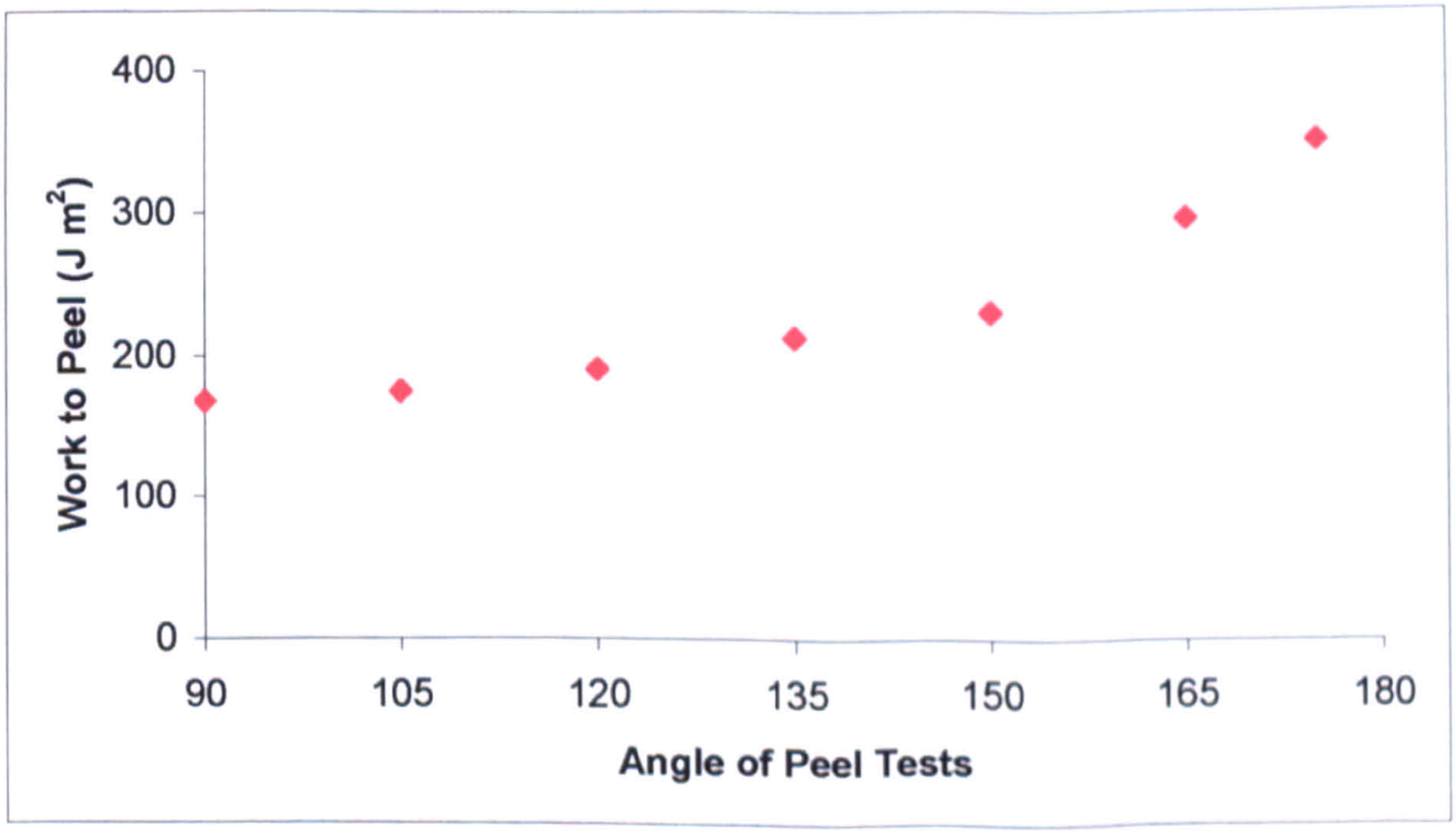


Fig. 10 b. An example of the typical effect of the angle of the peel test on work to Peel in hemp.

from 90° to the "boundary angle" α_b and Group 2 was formed by the rest of the peel test data, from the "boundary angle" α_b to 175°. All possible combinations of data were considered and linear regression analysis of the data in the two groups, fitting the standard regression equation (1) to each data subset, was carried out (Schroeder *et al.*, 1986; Berk, 2003):

$$W = \beta_0 + \beta_1 \alpha + \varepsilon, \qquad (1)$$

Where: W is the work to peel;

α is the peeling angle;

β_0 and β_1 are the regression coefficients;

ε is an error term having a normal distribution with zero mean and standard deviation σ.

The results of the data partitioning are shown in Fig. 11. As the boundary angle α_b increases, the standard deviation for Group 1, which was initially greater than for Group 2, decreases whereas for the Group 2 it initially decreases and then starts to increase. The determination coefficient, which shows the proportion of variability in work to peel accounted for by the peeling angle, reaches its maximum at the boundary angle of 105° for Group 2 ($r^2 = 0.261$). The determination coefficient for Group 1 remains insignificant throughout. Thus, the data analysis suggests that the peel test conditions can be divided into two types, according to the effect of peeling angle on the work to peel:

Group 1 with peel tests at angles between 90° and 105°, where angular position does not affect the work to peel, and Group 2 with peel tests at angles of between 120° and 175° where work to peel is proportional to the peeling angle.

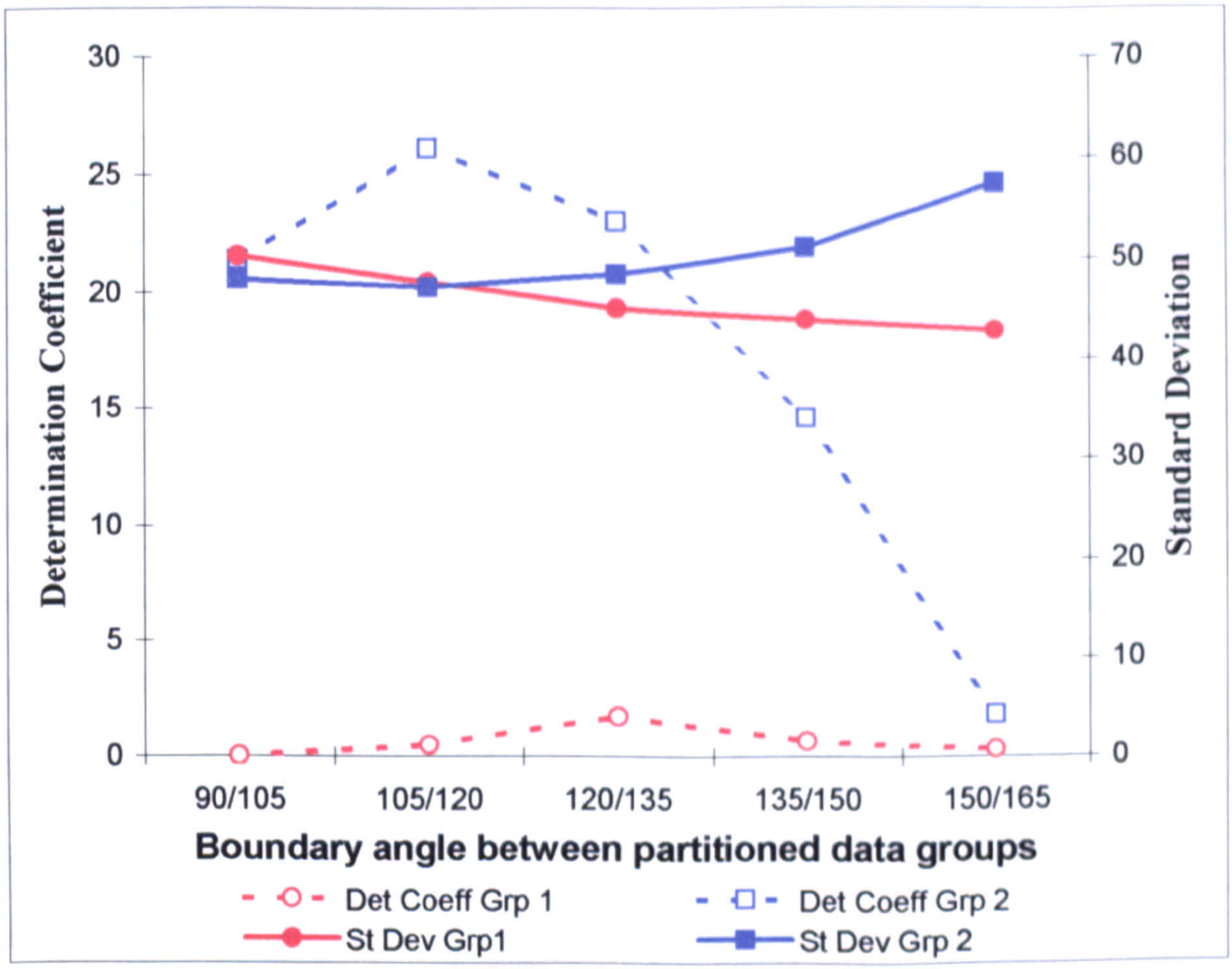


Fig. 11. Effect of the angle of the Peel Test on the work to peel in hemp: Data-partitioning and boundary angle.

Circles represent Group 1 (red), squares represent Group 2 (blue); solid lines represent standard deviation and dashed lines represent determination coefficient. The standard deviation for Group 2 is at minimum values for the boundary angle of 105° and begins to increase at a boundary angle of 120°. The determination coefficient for Group 2 reaches maximum at the boundary angle of 105°.

A boundary angle of 105° describes the data partitioning such that group 1 consists of data for peel tests carried out at 90° and 105°; and group 2 consists of data for peel tests carried out at 120°, 135°, 150°, 165° and 175°.

There was no significant correlation between peeling angle and work to peel in Group 1, with very low correlation coefficient and high probability of correlation being zero (r = -0.068, $P = 0.701$). This suggests that for peel tests between 90 ° and 105° the test conditions had no significant effect on the work to peel. For Group 2, comprising the data for peel tests between 120° and 175°, the correlation between peel angle and work to peel was significant, (r = 0.511, $P = 0.001$).

4.2.2 The effect of moisture content on work to peel in hemp (Appendix 4)

Peel test

Mean moisture content decreased from around 80% when fresh to around 12% after 17 hours in the oven at 40° C, samples were left in the oven for a further 25 hours to complete dehydration. Work to peel data from all individual stems shows considerable scatter (Fig. 12a), consideration of trends within the scatter of individual stem traces (Fig. 12b) indicates increasing work to peel as stem moisture content decreases below 30%. Work to peel then decreases rapidly as stems approach zero moisture content. Mean stem moisture content reflects this pattern (Fig. 12c) and examples of individual stems (Fig. 12d) indicates that work to peel reaches a peak and then begins to decrease rapidly as mid-stem moisture content falls below 20%.

Analysis of the variability in the data for individual stem's work to peel values at different stem moisture contents during dehydration in the oven shows substantial variability between 40% and 70% stem moisture content. Variability increases again as stem moisture content decreases below 20% and is greatest as stems become dry (Fig.13).

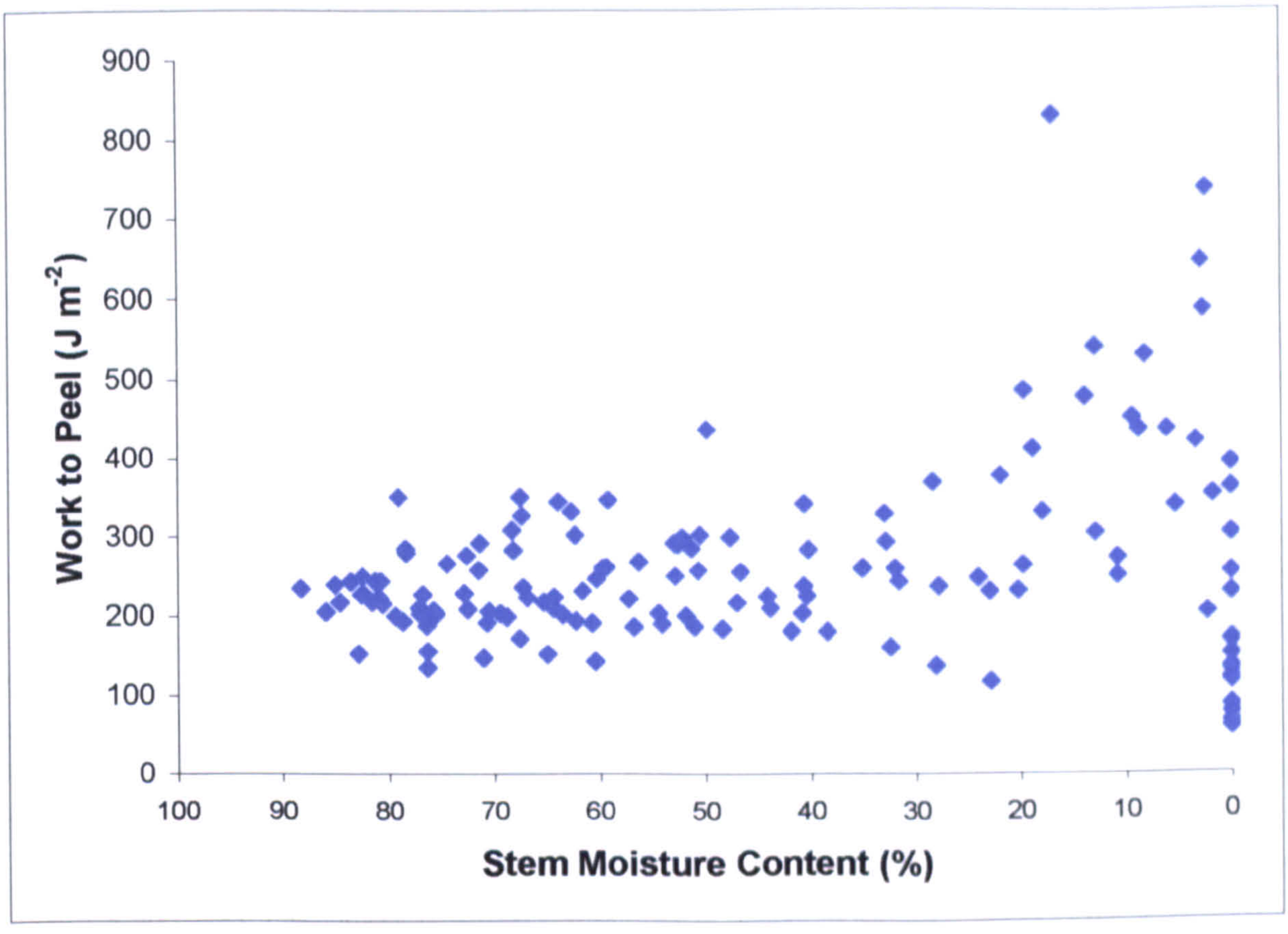


Fig. 12 a. The effect of stem moisture content on work to peel in oven-dried hemp: all data.

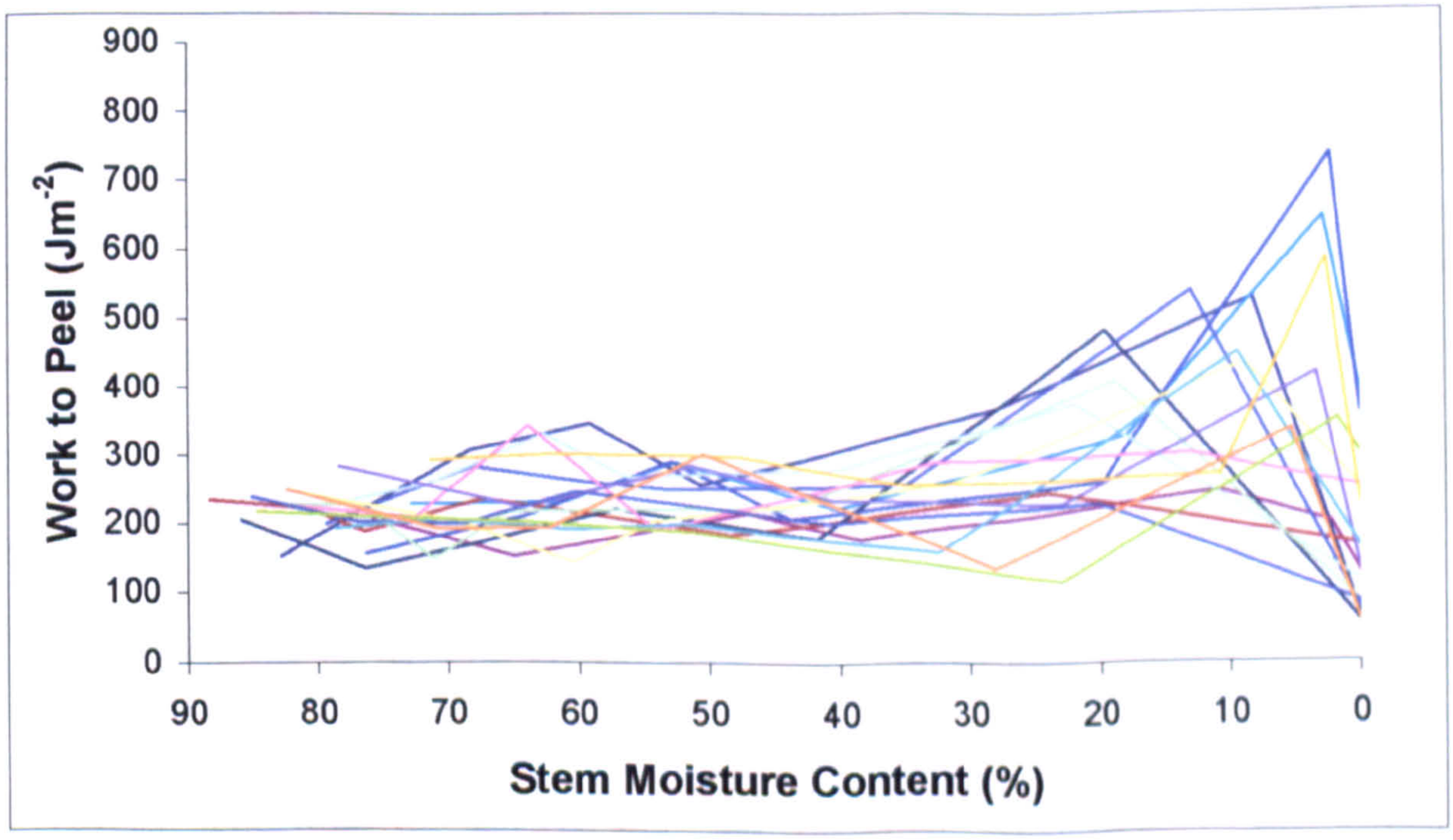


Fig. 12b. Work to peel for individual stems at different moisture content in oven-dried hemp.

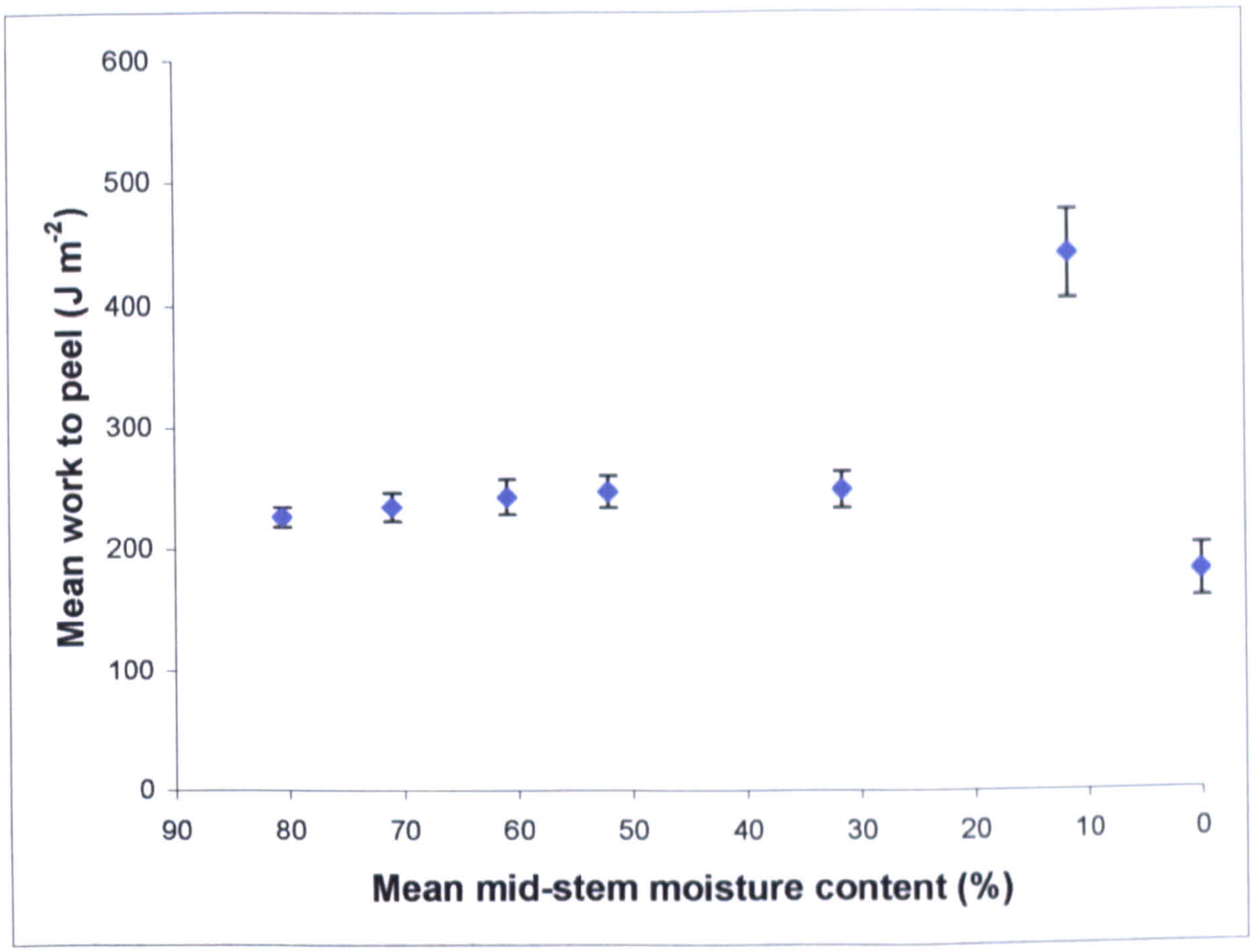


Fig. 12 c. The effect of stem moisture content on work to peel in oven-dried hemp.

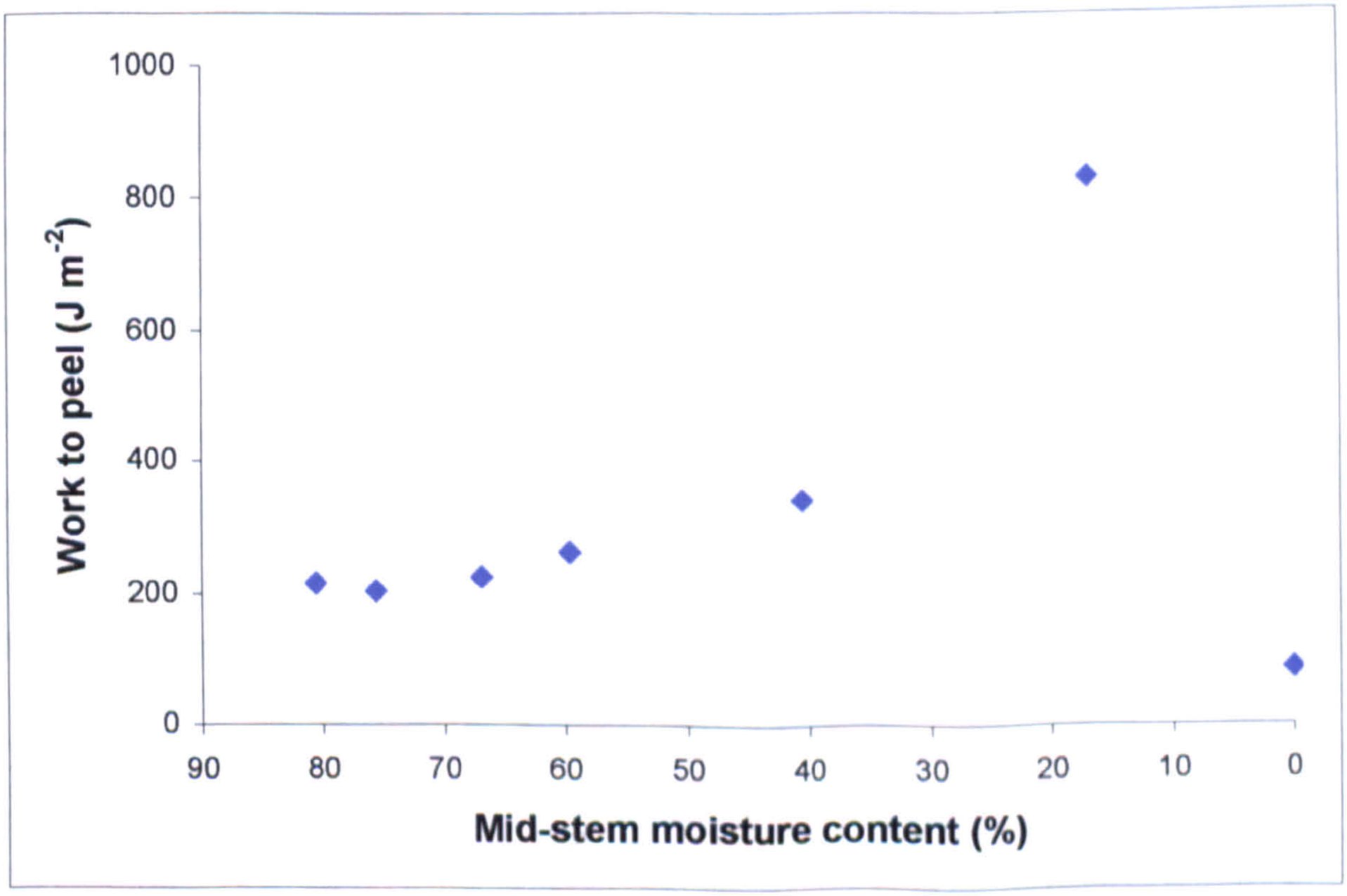


Fig 12 d. An example of the effect of stem moisture content on work to peel in oven-dried hemp stems. (stem no. 27).

As moisture content decreased, three distinct phases in the work to peel were identified. First, as mean moisture content (Fig. 12c) decreased from 80 to 30%, there was no significant change in work to peel ($P > 0.05$, df =12), which remained nearly constant (226 $\pm$ 8.2 to 248 $\pm$ 14.8 J m^{-2}). Second, as mean moisture content decreased from 30% to around 12 %, there was a significant increase in work to peel from 249 $\pm$ 14.8 to 441 $\pm$ 35.9 J m^{-2} ($P < 0.001$, df = 12). Finally, as mean moisture content decreased below 12 % work to peel significantly decreased from 441 $\pm$ 35.9 to 181 $\pm$ 21.6 J m^{-2} ($P < 0.01$, df = 12), and this effect was evident in all of the stems tested (Fig 12b; see Fig. 12d for an example of and individual stem). Mean work to peel at very low moisture contents was less than when the stems were fresh.

Investigation of the individual stems' force versus displacement traces indicated a change in the nature of the fracture characteristics (Fig.14a). In fresh stems the traces are relatively "smooth" and applied force is relatively low. As stem moisture content decreases and the applied force increases and the traces become increasingly variable, until stem moisture content reaches around 20%. As stem moisture content decreases below 20%, the applied force begins to decrease and as the stems' moisture contents approach zero the trace again becomes "smooth". An example of an individual stem is shown in Fig.14b.

4.2.3 Progress of retting in stand-retted flax (University Farm) (Appendix 5)

Desiccation of the stems

Immediately prior to application of the glyphosate, the stems had a moisture content of around 62% and this decreased to around 10% during the course of the study (around 8 weeks). Over the whole experiment, both herbicide-treated and untreated control stems behaved in a similar manner (Fig. 15a). However, there

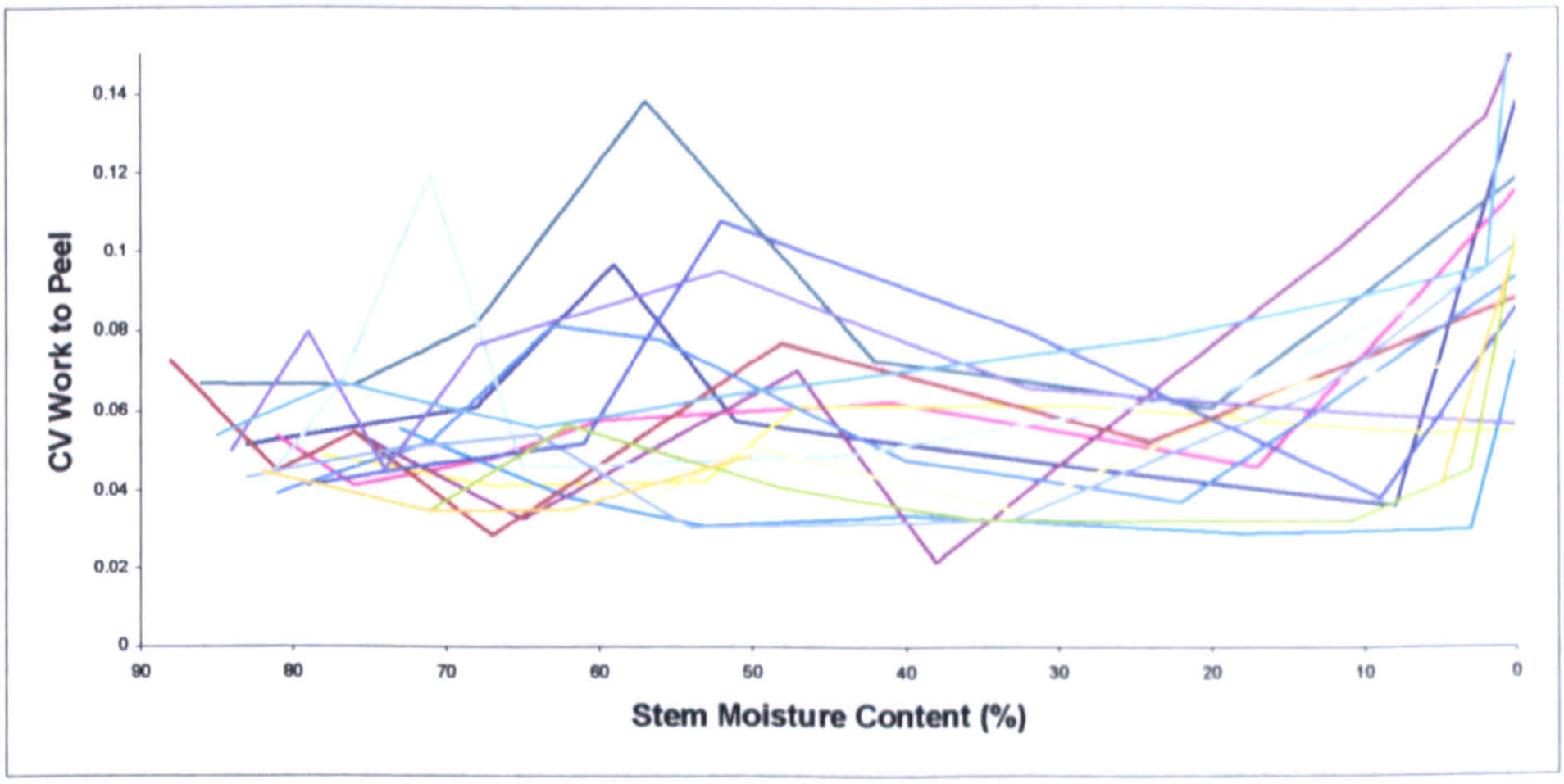


Fig. 13. Variation in individual stem's work to peel data at different moisture contents in oven-dried hemp.

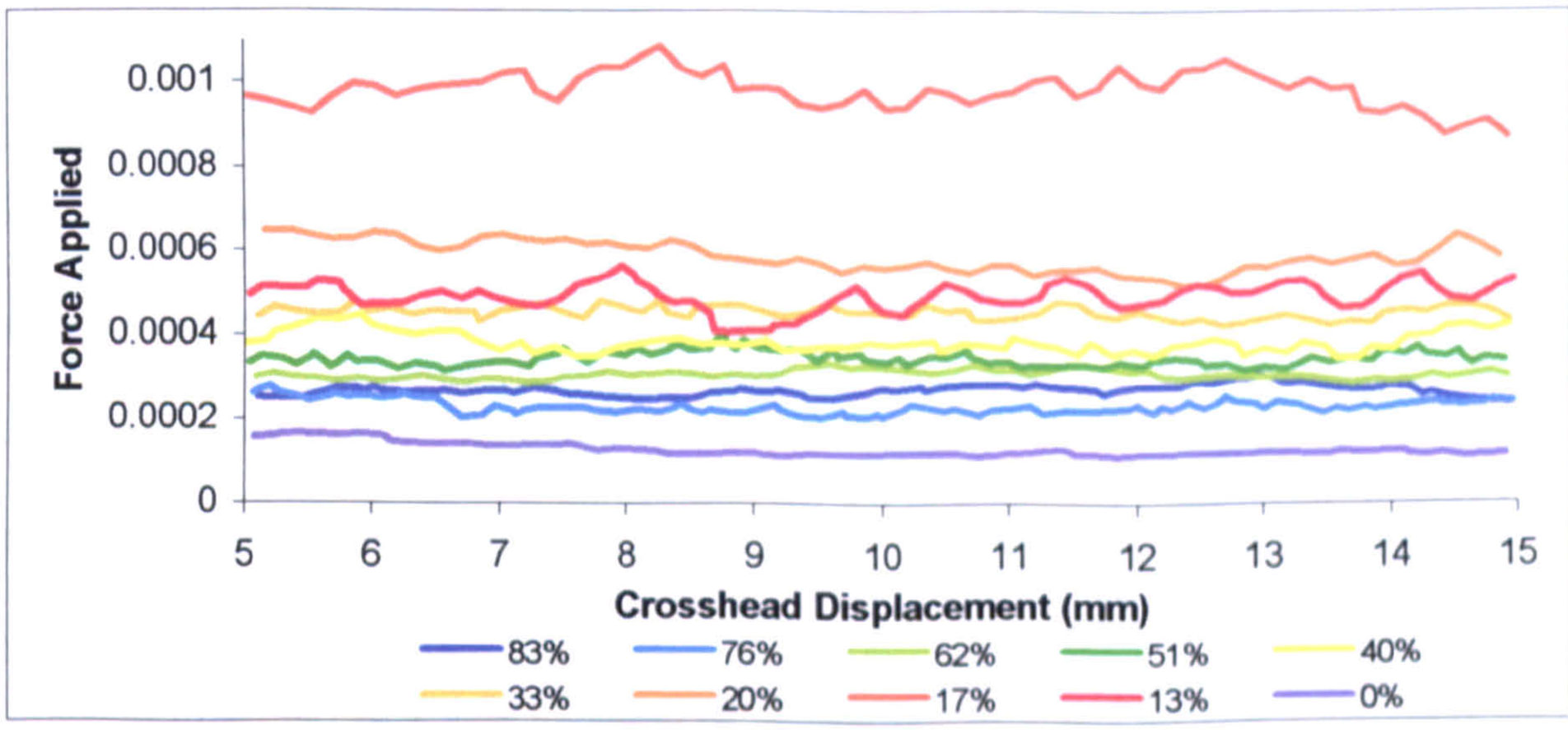


Fig. 14a. Typical force versus displacement traces, at a range of stem moisture contents, in oven-dried hemp peel tests.

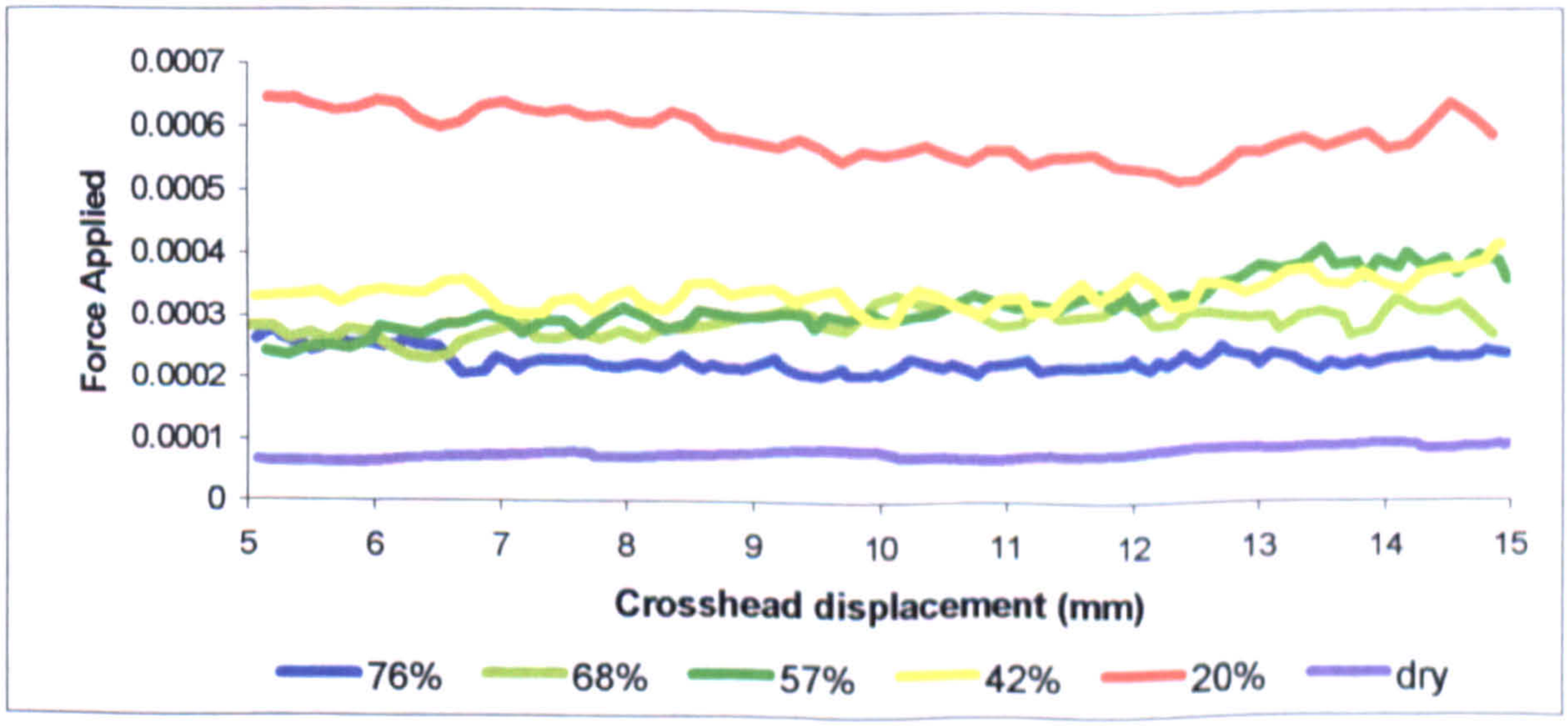


Fig. 14b. Examples of force versus displacement traces in peel tests on an individual oven-dried hemp stem, at a range of moisture contents (plant 17).

were differences between the treatments, the untreated stems actually began to dehydrate before the treated stems. The decrease in the moisture content of the stem was most marked between 12 and 19 days after the application of the glyphosate, with the rate of dehydration being more rapid in the treated stems. During this period, the moisture content of the treated stems decreased from 58 $\pm$ 0.9% to 13 $\pm$ 1.2% ($P < 0.01$, d.f. = 18) and in the untreated stems it decreased from 52 $\pm$ 1.0% to 20 $\pm$ 1.9% ($P < 0.01$, d.f. = 18). The moisture content of both treated and untreated stems decreased to around 11% by 32 days after application of the glyphosate and then stabilised around this level, falling below 10% only at 60 days after treatment.

Peel test

From the results of the peel test, three main phases in the work to peel can be identified over the course of the study; initially there is a short period of relatively consistent work to peel, followed by a rapid increase in work to peel between 12 and 19 days after treatment and then a period of decreasing work to peel (Fig. 15b).

For the first five days in untreated stems and the first twelve days in glyphosate-treated stems, the work to peel remained relatively constant around 160 to 170 J m^{-2}. Between 12 and 19 days after treatment there was a large, significant increase in the work to peel; in the glyphosate-treated stems it increased to 549 $\pm$ 17.8 J m^{-2} ($P < 0.01$, df = 18) and in the untreated stems it increased to 475 $\pm$ 25.1 J m^{-2} ($P < 0.01$, d.f. = 18) (Fig. 15b). There was a significant difference between the two treatment's maximum recorded work to peel (at 19 days). The maximum work to peel was 16% greater for the treated stems than for the untreated stems ($P < 0.05$, d.f. = 18). This increase in work to peel corresponded with a very rapid

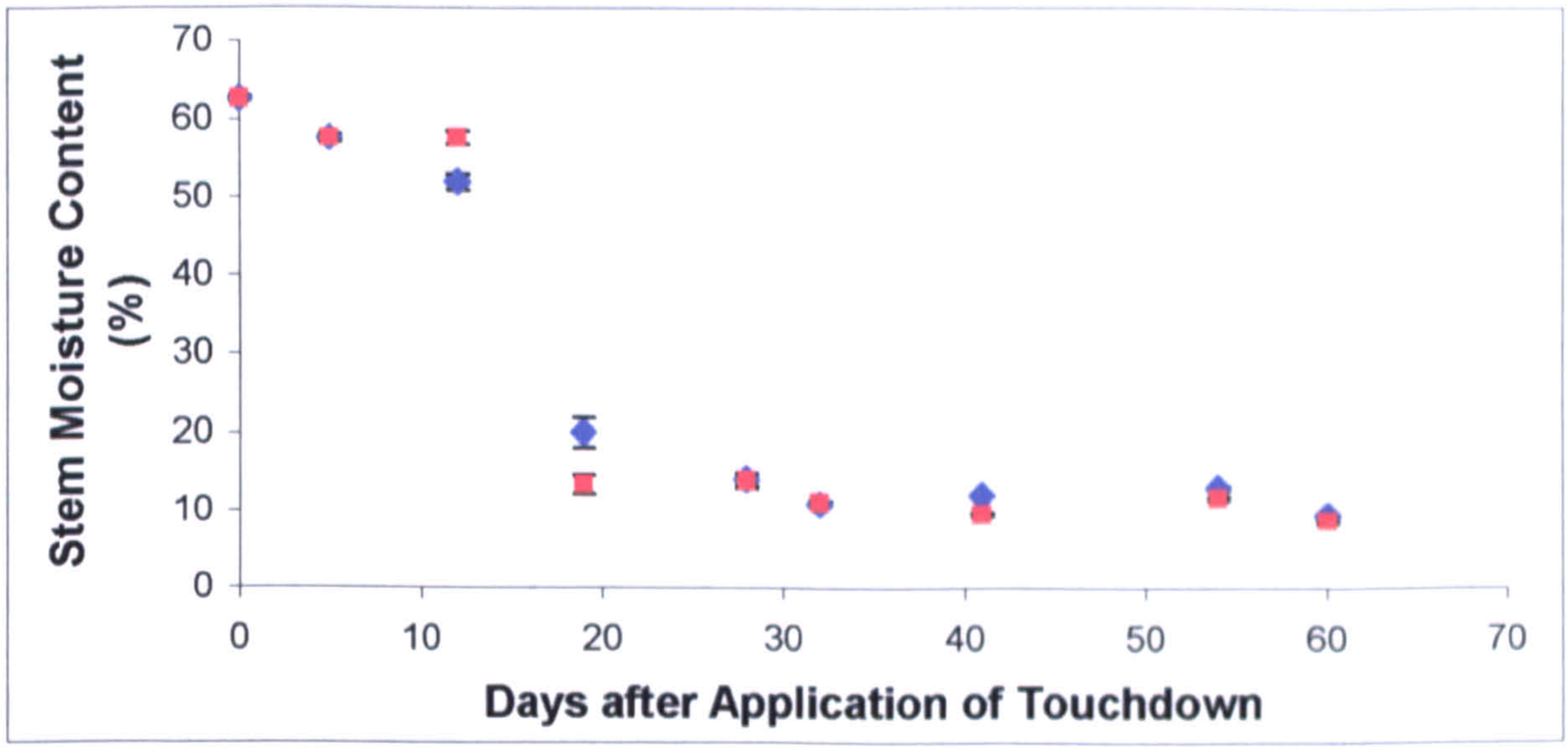


Fig. 15a. Progress of desiccation around the mid-point of the stem in stand-retted flax at De Montfort University Farm; untreated control (♦) and Touchdown-desiccated (■).

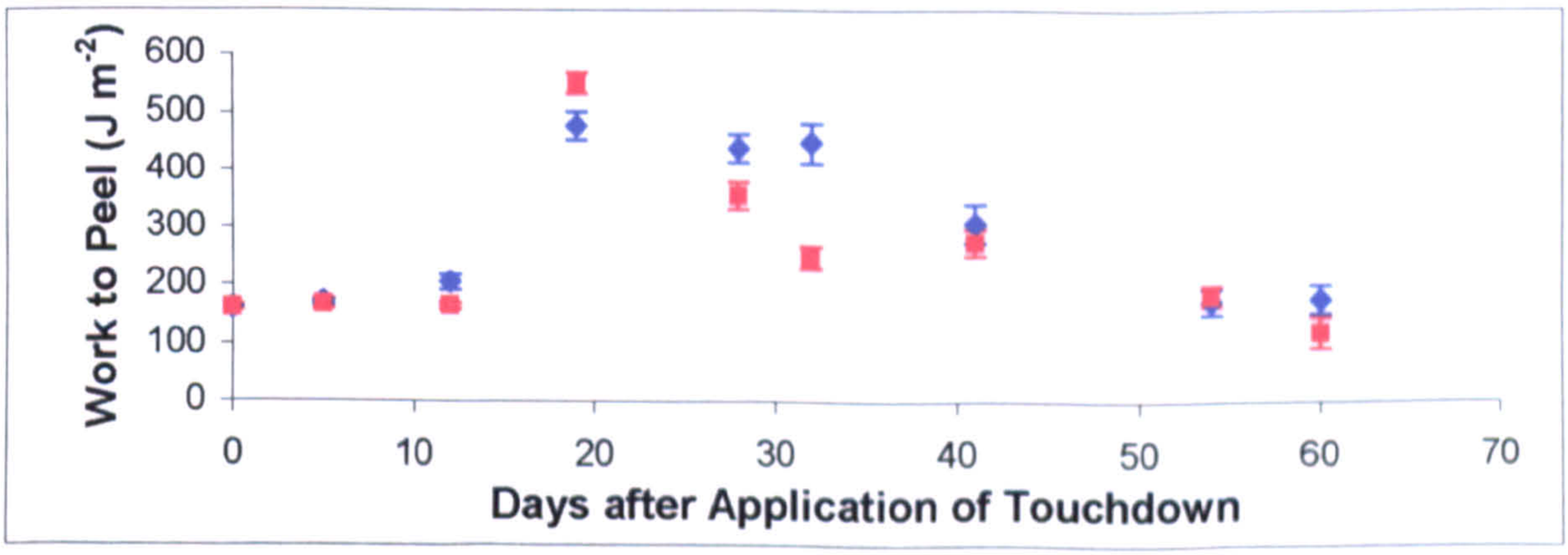


Fig. 15b. Changes in work to peel over the retting period in desiccated, stand-retted flax at De Montfort University Farm; untreated control (♦) and Touchdown-desiccated (■).

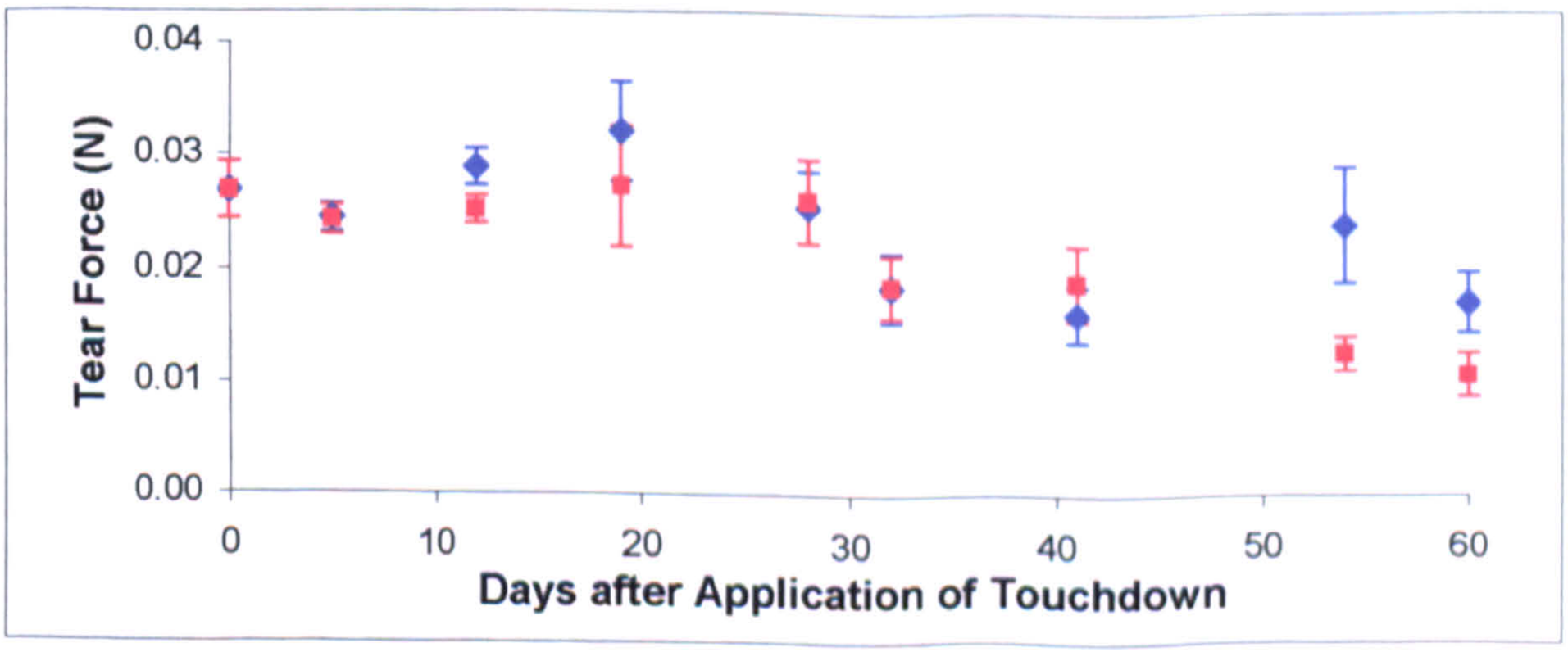


Fig. 15c. Changes in trouser-tear force over the retting period in desiccated, stand-retted flax at De Montfort University Farm; untreated control (♦) and Touchdown-desiccated flax (■).

dehydration of the treated stems (Fig. 15a). After 19 days the work to peel decreased again, but there were significant differences between the two treatments (Fig. 15b). In treated stems there was an immediate significant decrease in the work to peel, from 549 $\pm$ 17.8 J m^{-2} down to 248 $\pm$ 19.1 J m^{-2} ($P < 0.01$, d.f. = 18), but in the untreated stems the work to peel initially remained unchanged (for a further 13 days), it was only then that the untreated stems showed a similar decrease in work to peel. After 54 days the mean work to peel for both treatments had dropped to around 170 to 180 J m^{-2}. Thereafter, by 60 days after treatment, the work to peel for treated stems had decreased to 117 $\pm$ 27.2 J m^{-2}, while the work to peel for untreated stems remained relatively constant, around 175 J m^{-2}.

Regression analysis showed strong negative correlation between moisture content and work to peel in both the untreated stems ($r^2 = 0.75$) and in the treated stems ($r^2 = 0.83$) (Fig. 16). Between 12 and 19 days after treatment with glyphosate there was a significant increase in the work to peel (Fig. 15b) corresponding with a significant decrease in the moisture content (Fig. 15a).

These results support those reported by Goodman *et al.* (2002). In their study the work to peel fresh stems was around 200 J m^{-2}, rising to 539 J m^{-2} at around 3 weeks after desiccation and decreasing to 297 J m^{-2} after around 7 weeks. In this study the work to peel at the 7 week stage was around 250 J m^{-2}. The work to peel data for untreated flax stems showed substantial variability between 30% and 40% stem moisture content and also at stem moisture contents below 15%. The work to peel data for desiccated, stand-retted stems showed similar variability (Fig. 17a).

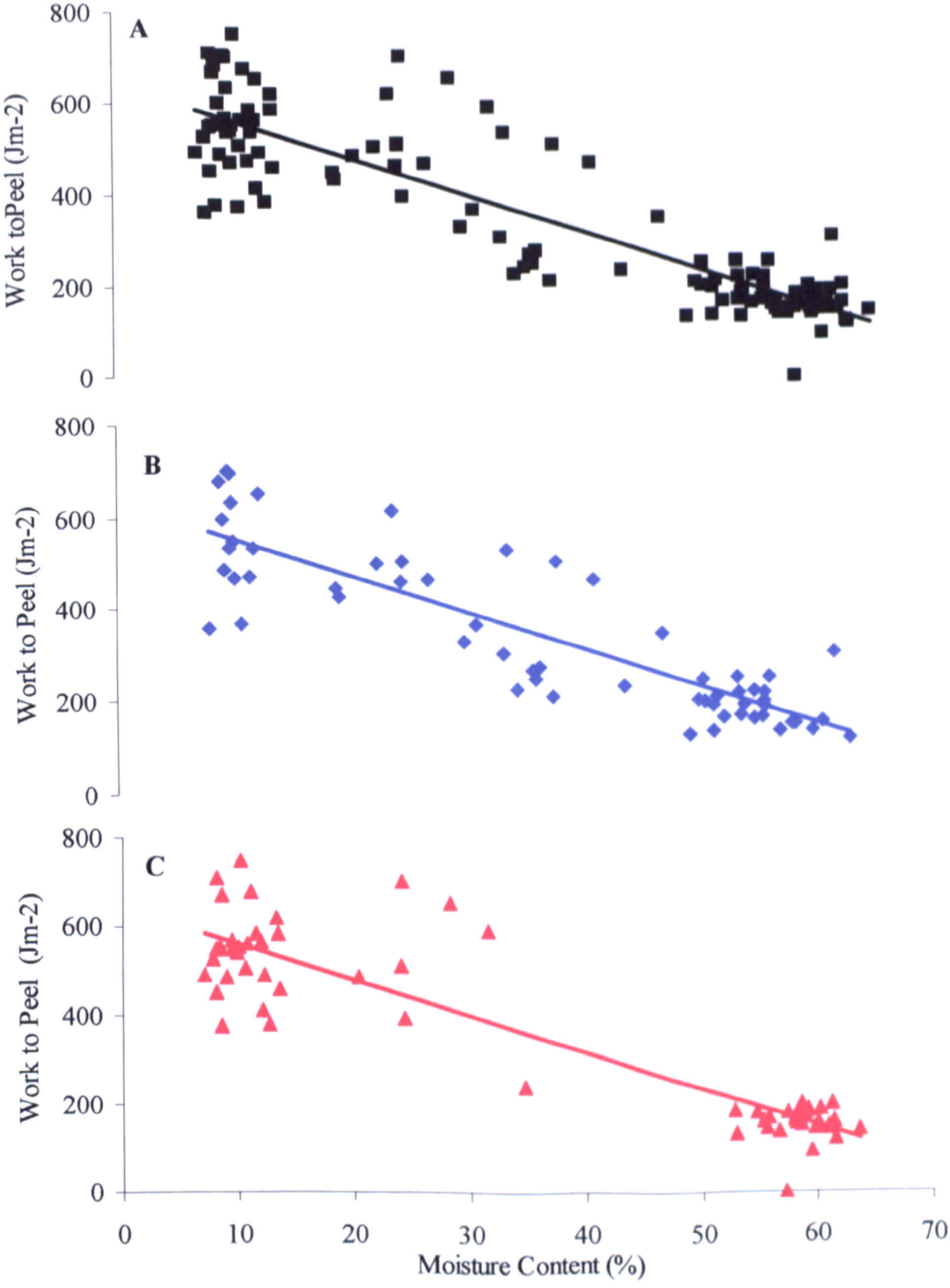


Fig. 16. The relationship between moisture content and work to peel. (12-19 days).
Regression analysis showed strong negative correlation between moisture content and work to peel. **A.** All treatments (■), $r^2 = 0.80$, **B.** Untreated (♦), $r^2 = 0.75$, and **C.** Touchdown (glyphosate) (▲), $r^2 = 0.83$.

Tear test

The force required to propagate the fracture in the tear test was smaller and more variable than that required in the peel tests. Analysis of variance showed no relationship between tear force and treatment, duration of retting period or moisture content (Fig. 15c), however, the trend was for the tear force to decrease over time. These results are similar to those reported by Goodman *et al.* (2002). The tear force data generally showed greater variability; analysis showed that the variability was greater at stem moisture contents below 25% (Fig. 17b).

4.2.4 The progress of retting in pulled and dew-retted hemp (Appendix 6)

Dehydration of the stems.

There was a significant difference in stem moisture content at pulling ($P < 0.01$, df = 12) with mid-stem moisture content of 79 ± 1.0% for Kompolti and 76 ± 0.9% for Tiborszallasi. After pulling, during the retting period, the decrease in stem moisture content was significant for both varieties; over a period of 34 days it decreased from around 80% to 11% for Kompolti and from around 75% to 10% for Tiborszallasi ($P < 0.01$, df = 12) (Fig. 18b).

Over the retting period there was a marked change in colour of the stems, as they senesced and then retted. The freshly harvested stems were green, changing to yellow and eventually almost white. Following heavy rain, around 21 days after pulling, the stems became contaminated with soil splashed from the ground and there appeared to be a rapid increase in colonisation of the stems by fungi shortly afterwards.

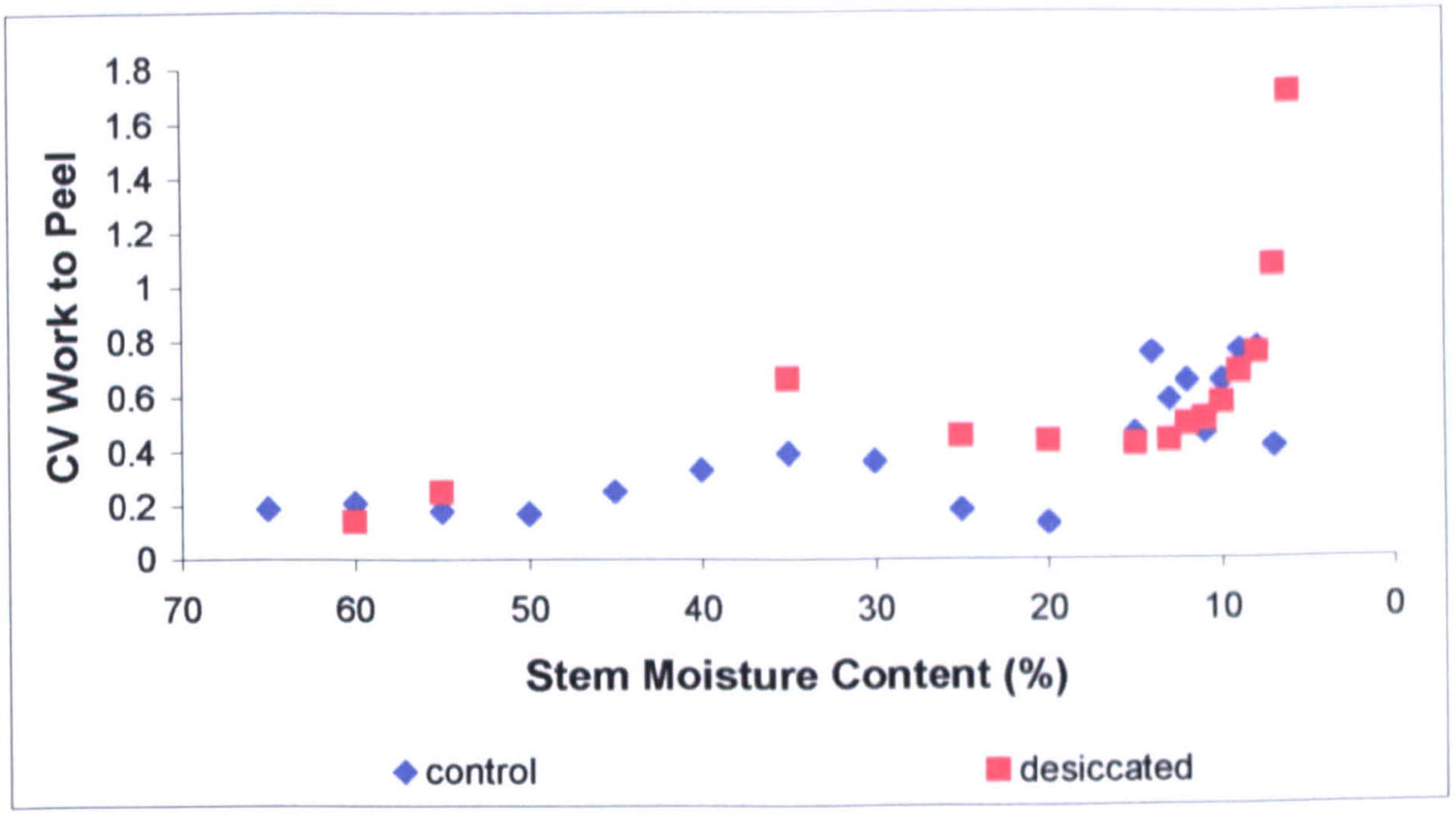


Figure 17a. Variability of work to peel in flax

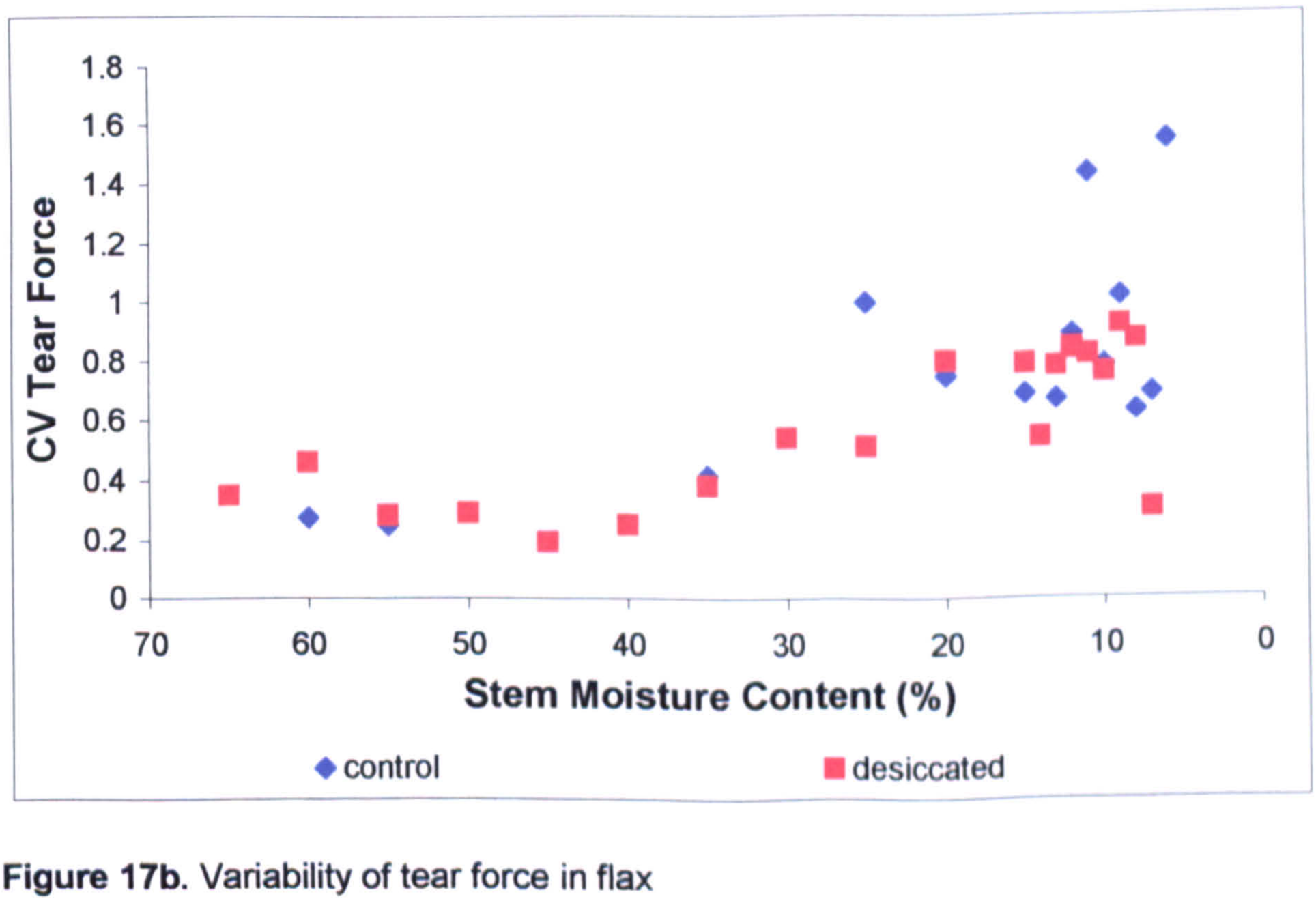


Figure 17b. Variability of tear force in flax

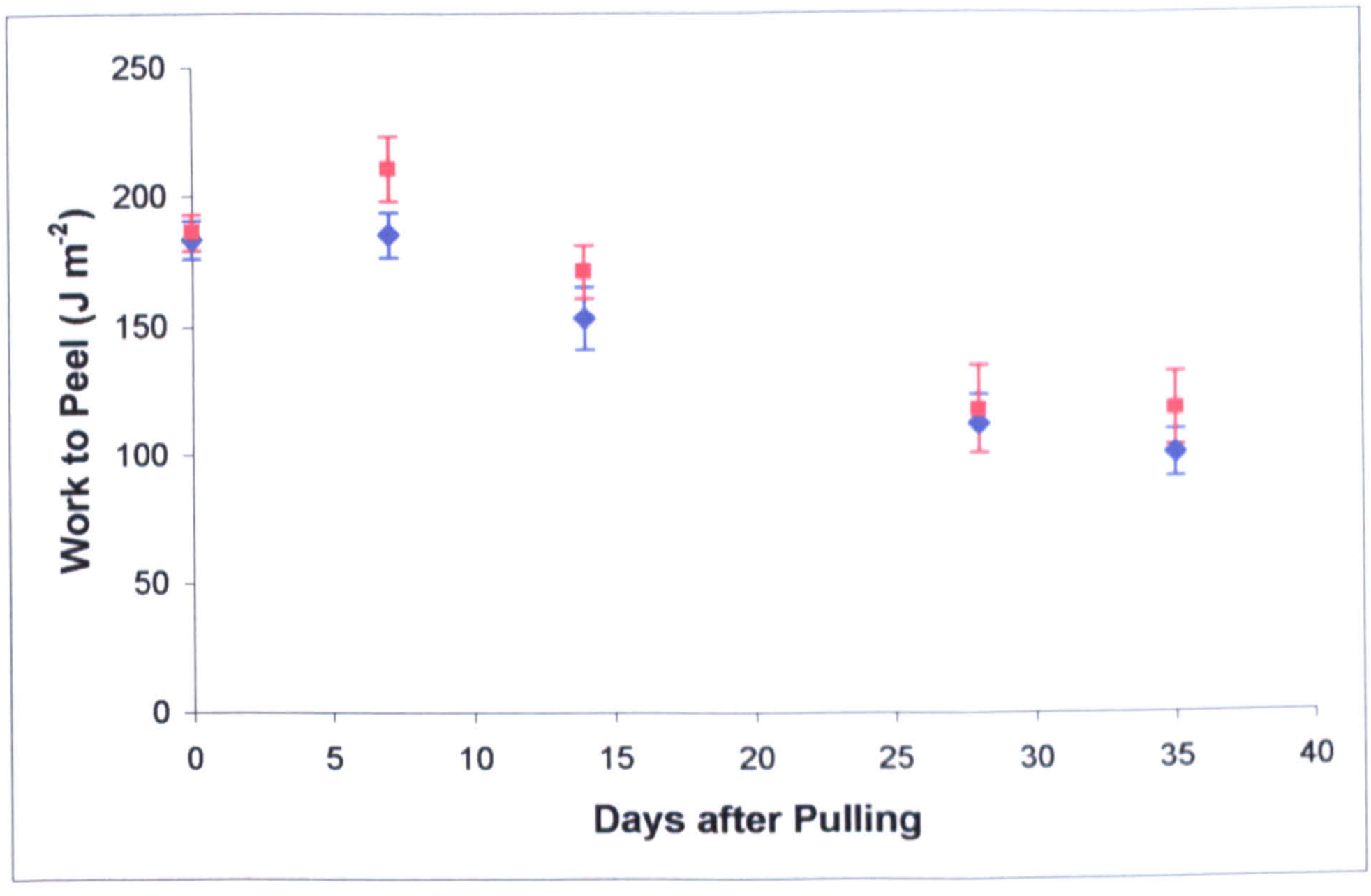


Fig 18a. The effect of dew-retting on the work to peel in hemp, Kompolti (◆) and Tiborszallasi (■).

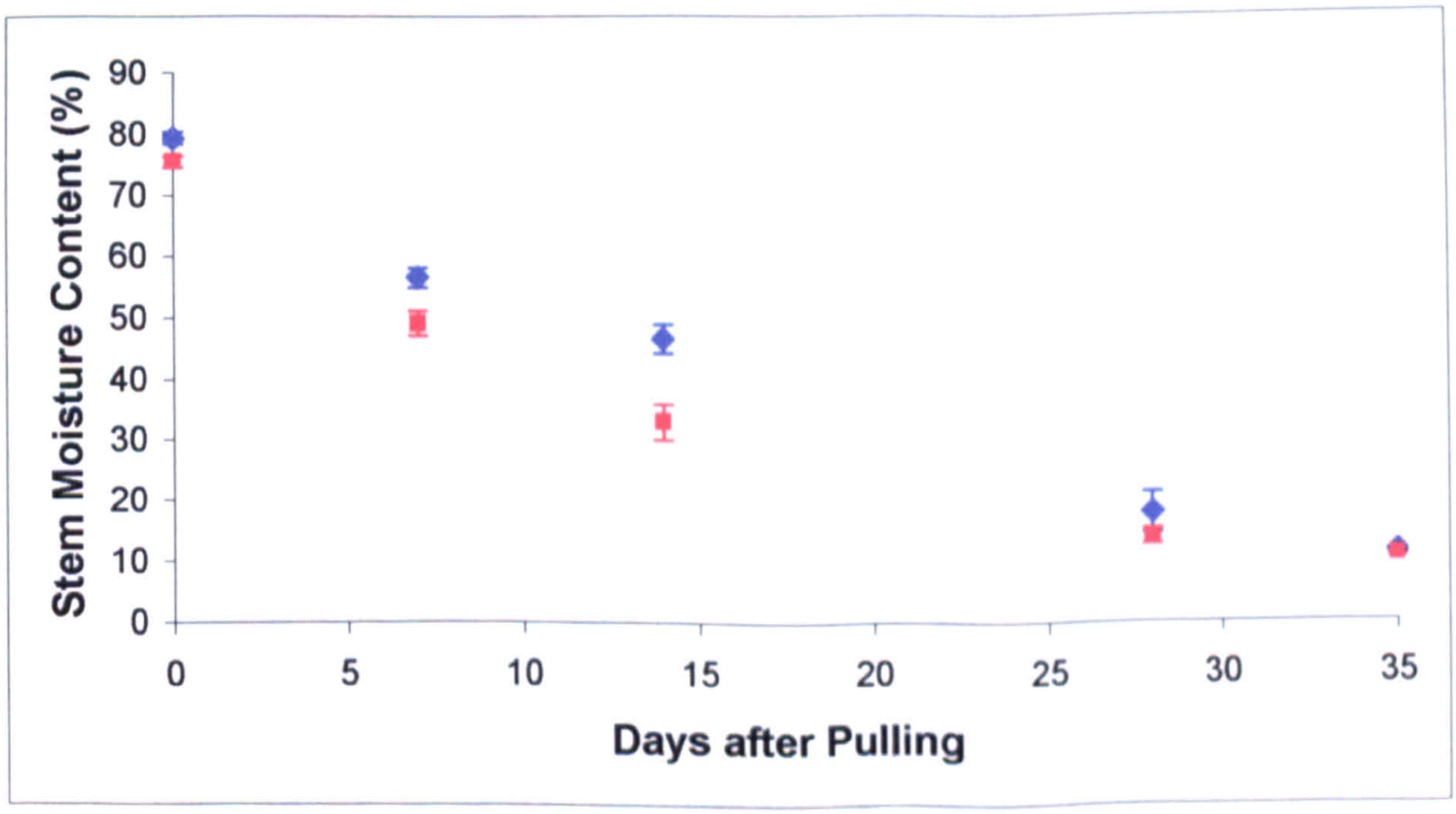


Fig. 18b. The decrease in moisture content around the mid-point of the stem in dew-retted hemp, cvs Kompolti (◆) and Tiborszallasi (■).

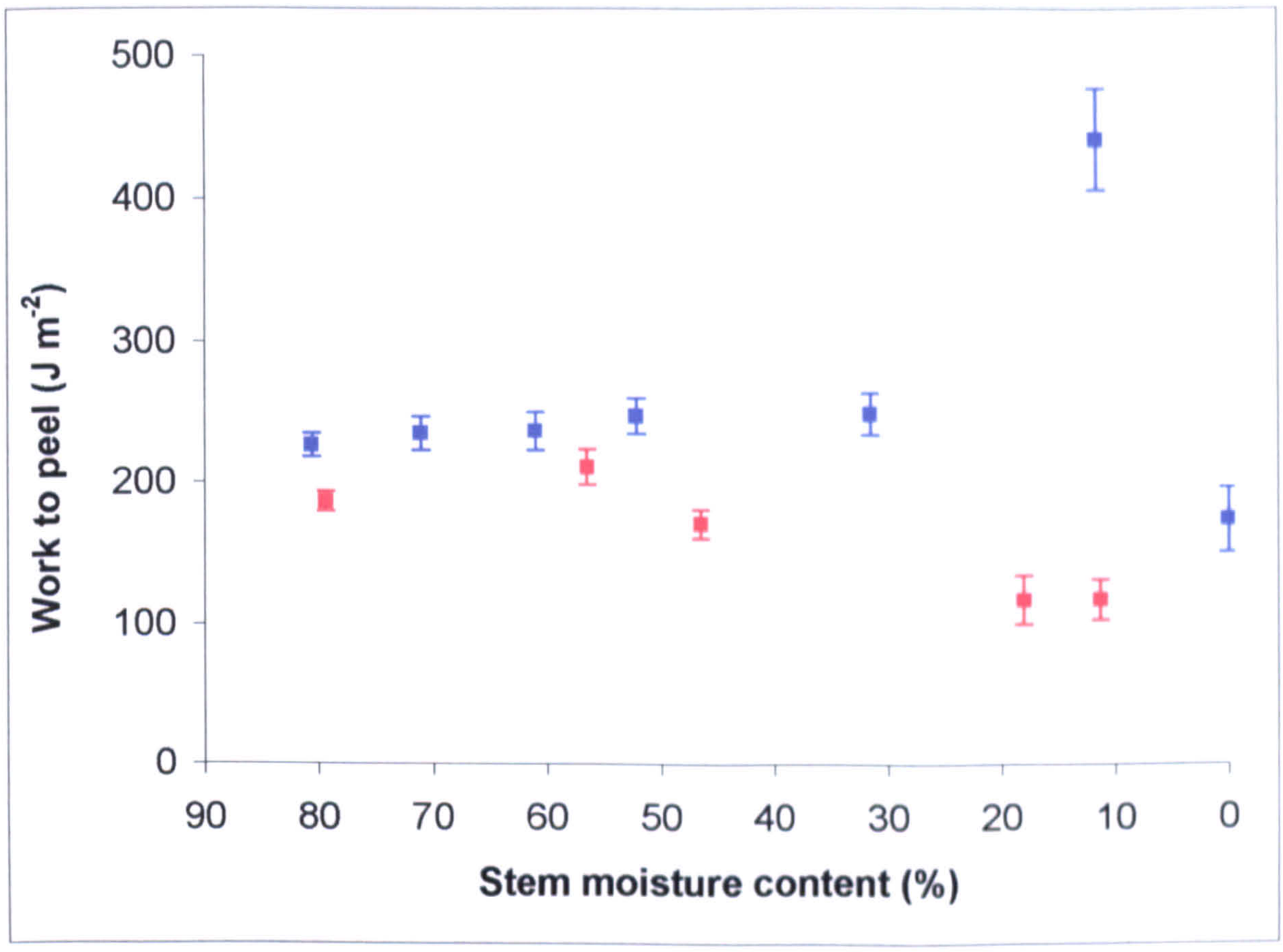


Fig. 19. A comparison of the work to peel in oven-dried (■) and dew-retted (■) hemp stems (cv. Kompolti).

Peel test

Once the stems were pulled and laid out on the ground to ret, they dehydrated and work to peel decreased; during the 34 days of dew-retting there was a significant decrease in stem moisture content in both varieties from around 80% to around 10% ($P < 0.01$, df = 12) (Fig. 18b). In the first 14 days after pulling, stem moisture content decreased to around 50%, but there was no significant change in work to peel, which decreased from 185 J m^{-2} to around 160 J m^{-2} ($P > 0.05$, df = 12). However, between 14 and 35 days after pulling, as stem moisture content decreased from 50 to 10% ($P < 0.05$, df = 12) (Fig. 18b), there was a significant decrease in work to peel, from 170 $\pm$ 10.1 J m^{-2} to 118 $\pm$ 14.2 J m^{-2} in Kompolti and from 153 $\pm$ 11.9 J m^{-2} to 100 $\pm$ 9.3 J m^{-2} in Tiborszallasi ($P < 0.05$, df = 12) (Fig. 18a). When work to peel was plotted against moisture content rather than time (Fig.19), a comparison could be made with the oven-dried samples.

4.2.5 The ease of decorticatio of enzyme-retted flax following increasing periods of time in a retting solution (Appendix 7)

In the first experiment, stems were decorticated on the inclined plane decorticator after increasing lengths of time in the retting solution. Results showed no firm relationship between the length of time that the sample was in the retting solution (the duration of the retting period) and the proportion of shive removed (Fig. 20a).

In a second experiment, the loose but entangled shive was removed after each decortication pass by applying a low velocity longitudinal air stream from a suction pump. The results from this experiment show a significant relationship between the duration of the retting period and the ease of decortication ($P < 0.05$).

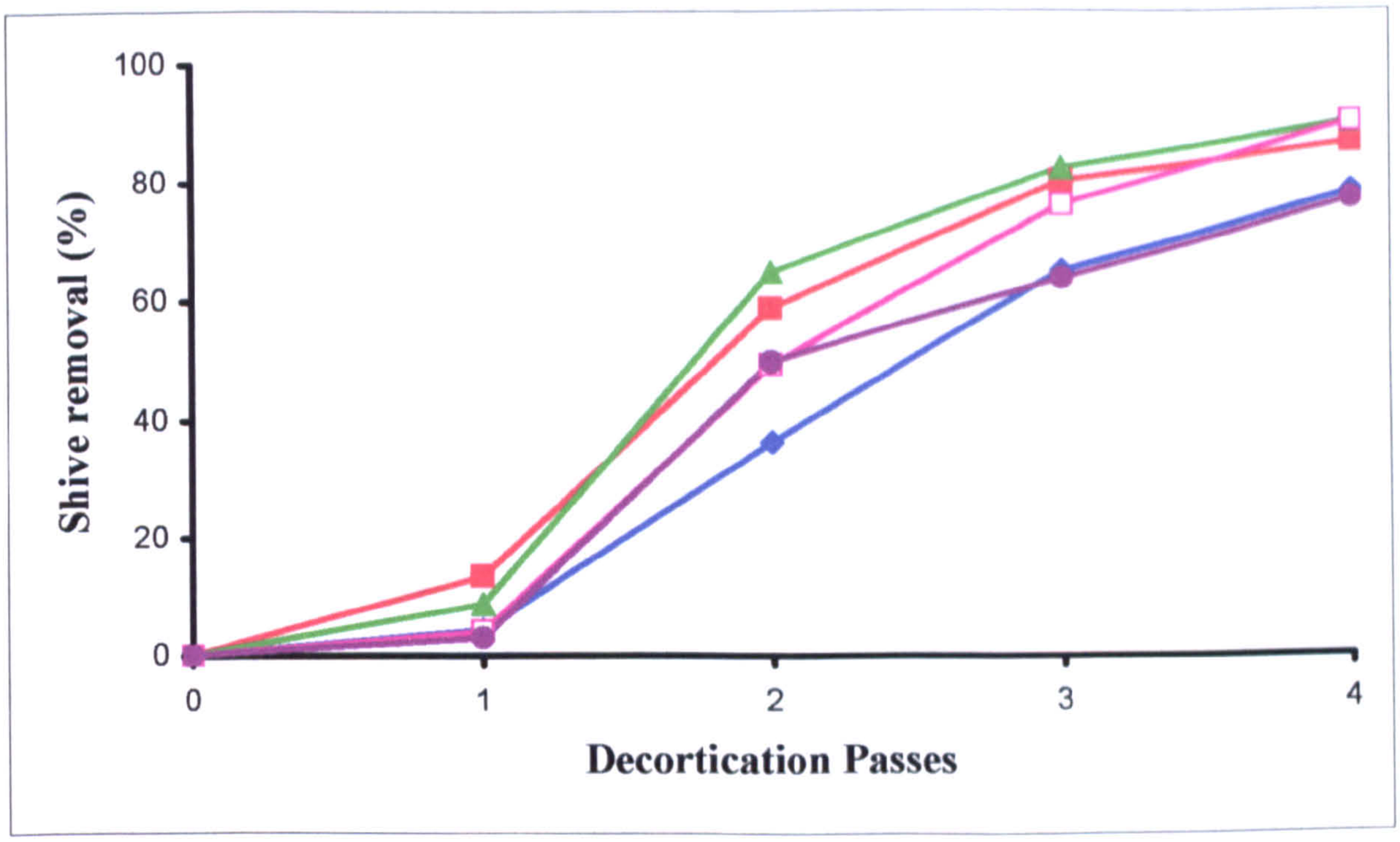


Fig 20a. Decortication of enzyme-retted flax with no secondary airstream shive removal system, after increasing periods of time in the retting solution.
Retting duration: 16 hrs (♦), 20 hrs (■), 24 hrs (▲), 40 hrs (□), 44 hrs (●).

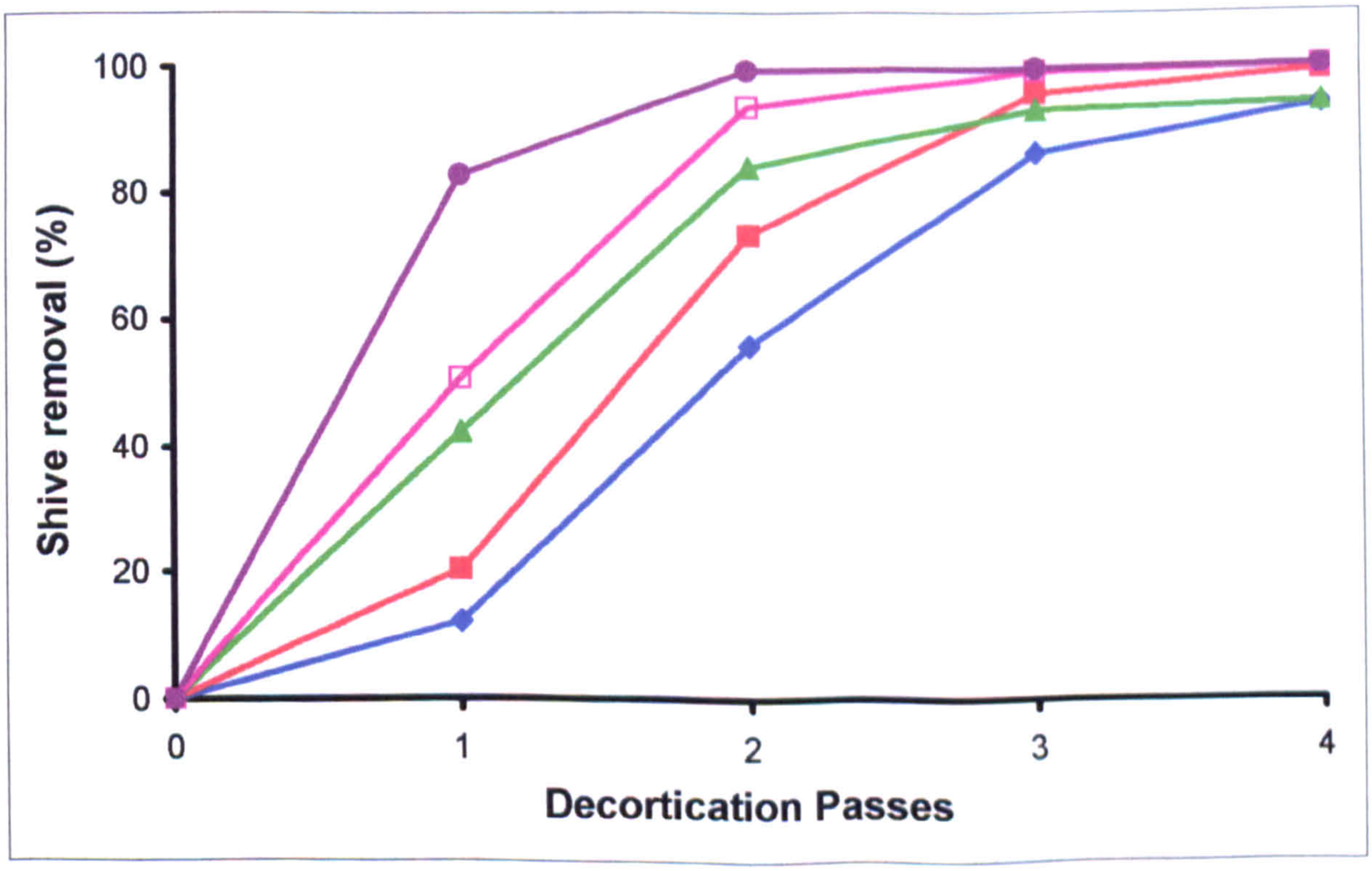


Fig 20b. Decortication of enzyme-retted flax with secondary airstream shive removal system, after increasing time in the retting solution.
Retting duration: 16 hrs (♦), 20 hrs (■), 24 hrs (▲), 40 hrs (□), 44 hrs (●).

The proportion of shive removed by each decortication pass increased with the duration of the retting period (Fig. 20b). For example, after 16 hours retting, a single decortication pass removed around 12% of the shive while after 44 hours retting a single decortication pass removed over 80% of the shive. After 16 hours retting, two decortication passes removed over 50% of the shive, while after 44 hours retting they removed over 99% of the shive. Thus the first two decortication passes differentiated between treatments showing the relationship between duration of the retting period and ease of decortication; as retting progressed the variability in results decreased (Fig. 21).

4.2.6 The ease of decortication of herbicide-desiccated, stand-retted flax (Huit farm) (Appendix 8)

Desiccation of stems

Immediately prior to application of the glyphosate, the stems had a moisture content of around 65% and this decreased to around 15% for the herbicide desiccated stems during the course of the study (around 11 weeks), while the untreated control stems remained around 60% throughout. (Fig.22). Dehydration was most marked between 15 and 29 days after the application of the glyphosate. During this period, there was a significant decrease in stem moisture content of the treated stems from 61% to 24% ($P < 0.01$). The moisture content of treated stems decreased below 20% by 43 days after application of the glyphosate and then stabilised around this level.

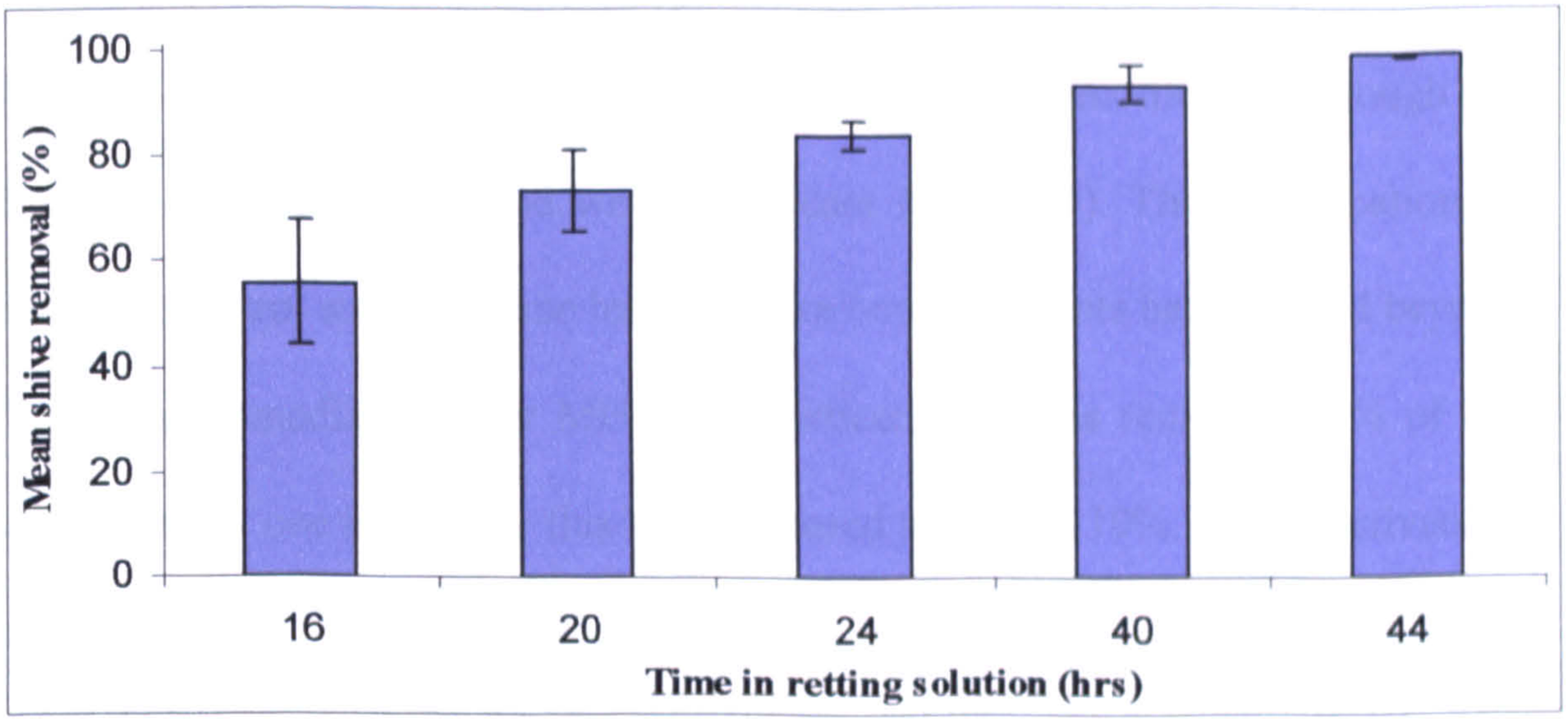


Fig. 21. Relative ease of decortication of enzyme-retted flax after 2 decortication passes.

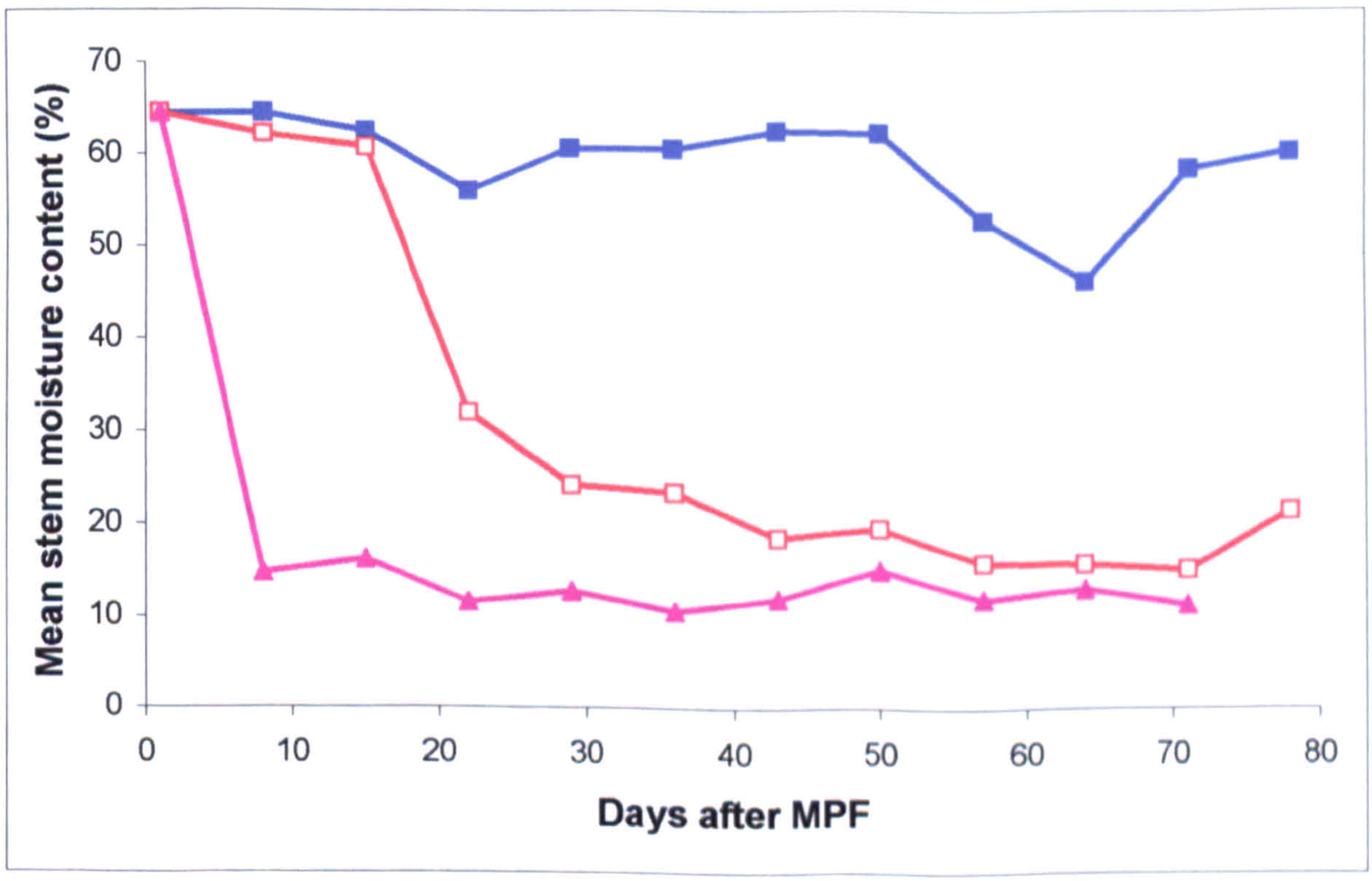


Fig 22. Desiccation of stems in stand-retted flax; Untreated control (■), Quattro desiccated (□) and pulled (▲) stems.

The Quattro-treated stems desiccated relatively slowly during the first 14 days after treatment, but then desiccated more rapidly down to around 20 % between 14 and 28 days after treatment and then remained relatively constant around that level thereafter. The pulled plants dehydrated very rapidly to 15 % within 7 days.

Ease of decortication

Analysis of variance showed that the changes in ease of decortication through the duration of the retting period were significant ($P < 0.05$). The decortication of untreated control stems became less effective once the plants had matured beyond flowering; immediately after MPF, 2 decortication passes removed 50% of the shive; but just one week later this had decreased to around 10%. The effectiveness of decortication then began to increase again, but was never as effective as it had been at MPF. At 6 - 8 weeks after MPF, 2 decortication passes removed only 20 - 30% of the shive; and even at 11 weeks after MPF, 2 decortication passes removed only 40% of the shive (Fig. 23). This effect was also observed to a greater extent in the desiccated stems and to a lesser extent in the pulled stems.

Following application of Quattro, decortication initially became less effective, the effect was greater than in the untreated controls, after 2 weeks 2 decortication passes removed only 5% of the shive and even by 6 weeks after treatment this had still only increased to around 15% shive removal. However, by 8 weeks after treatment, 2 decortication passes removed over 70% of the shive and then remained constant around this level to the end of the investigation (Fig. 23).

The stems that were pulled and stand-retted in the wire frames also exhibited the initial reduction in effectiveness of decortication, but it was less marked than in either the control or desiccated treatments. After 2 weeks around 35% of the shive was removed by 2 passes, but then by 4 weeks it was over 80%, by 6 weeks it was over 90% and by 8 weeks it was almost 100% shive removal (Fig. 23).

In a separate investigation, a large sample of flax stems was collected and the mid-stem diameters of 1200 stems were measured, to confirm the mean mid-stem

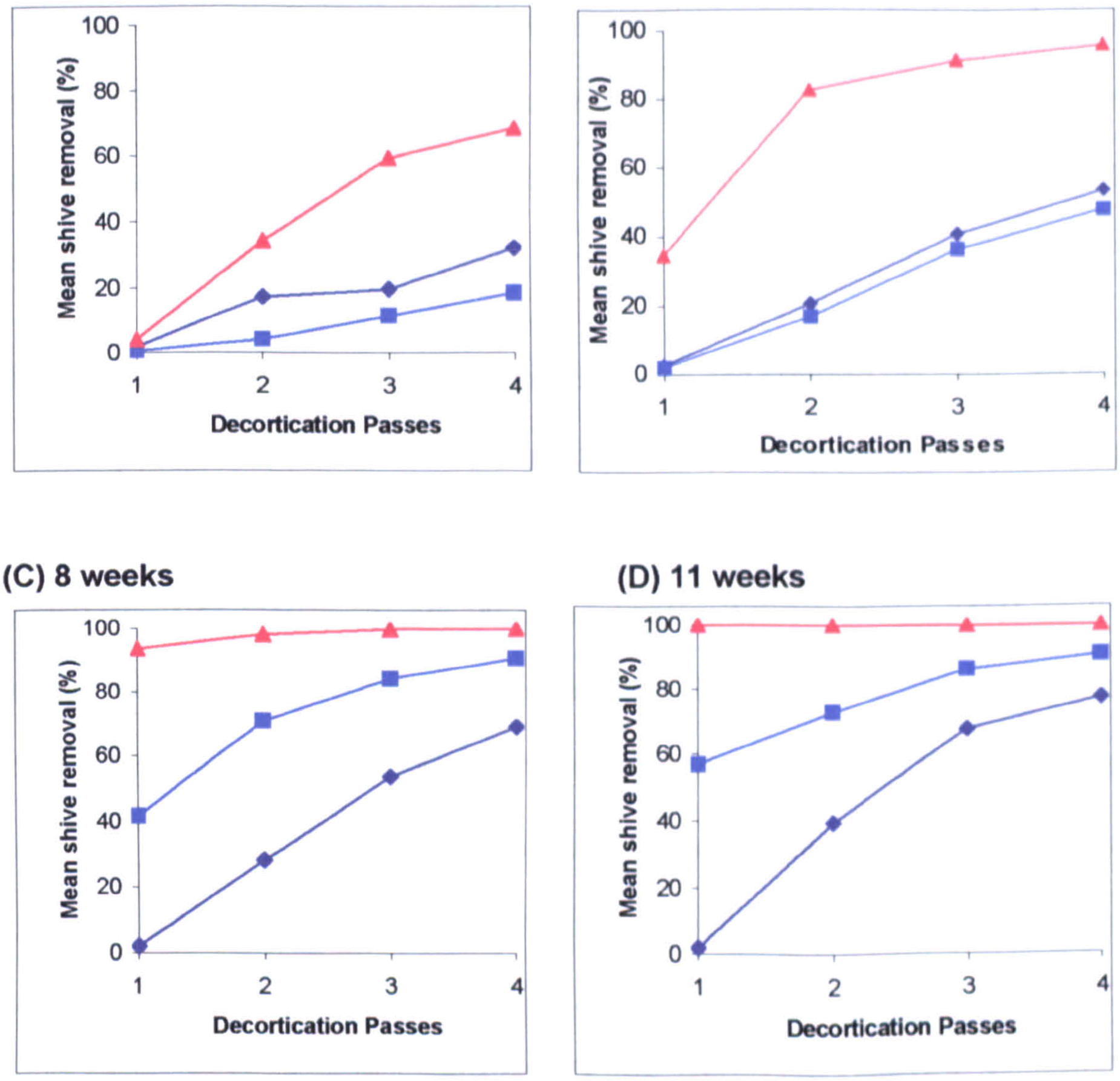


Fig. 23. Ease of decortication of desiccated, stand-retted flax straw from Huit Farm.

Ease of decortication tests were carried out after 2 weeks (A), 4 weeks (B), 8 weeks (C) and 11 weeks (D); untreated control (♦), Quattro desiccated (■) and pulled and stand-retted (▲) samples were evaluated using the inclined plane decortication test.

diameter of 1.93 mm. Samples were prepared from 30 stems, all with mid-stem diameter of 1.93 $\pm$ 0.10 mm and these were decorticated on the inclined plane decorticator. The resulting proportion of shive removed by each decortication pass was plotted separately rather than cumulatively to show the effectiveness of each individual decortication pass.

The first decortication pass in each series flattened and crimped the straw, but was relatively ineffective in terms of shive removal but the second decortication pass was more effective, differentiating between treatments (Fig. 24). The effectiveness of the subsequent third and fourth passes was dependent on the earlier passes, so could not be used as an indicator of the effectiveness of decortication since there had already been substantial changes to the structure of the stem. Thus, the second pass was most appropriate for making comparisons between treatments; this showed the decrease in the effectiveness of decortication immediately after MPF in the untreated control and a similar response in herbicide-treated stems (Fig. 25). Analysis of variance showed that the ease of decortication of the herbicide-treated stems was not significantly different to that of the untreated control stems, but the stems from pulled plants were significantly easier to decorticate than both the control and the herbicide-treated stems ($P < 0.05$).

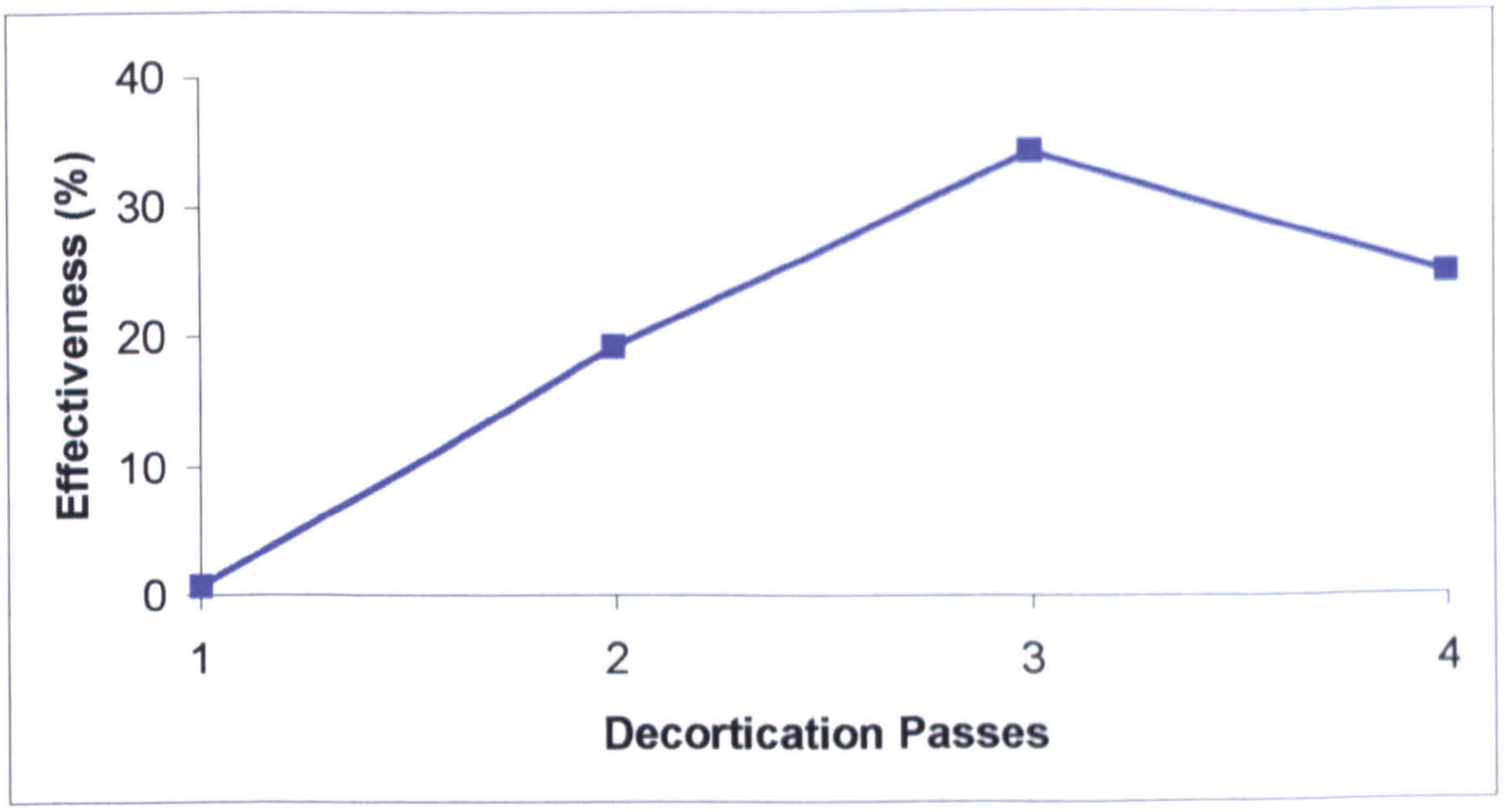


Fig 24. The effectiveness of each successive decortication pass.

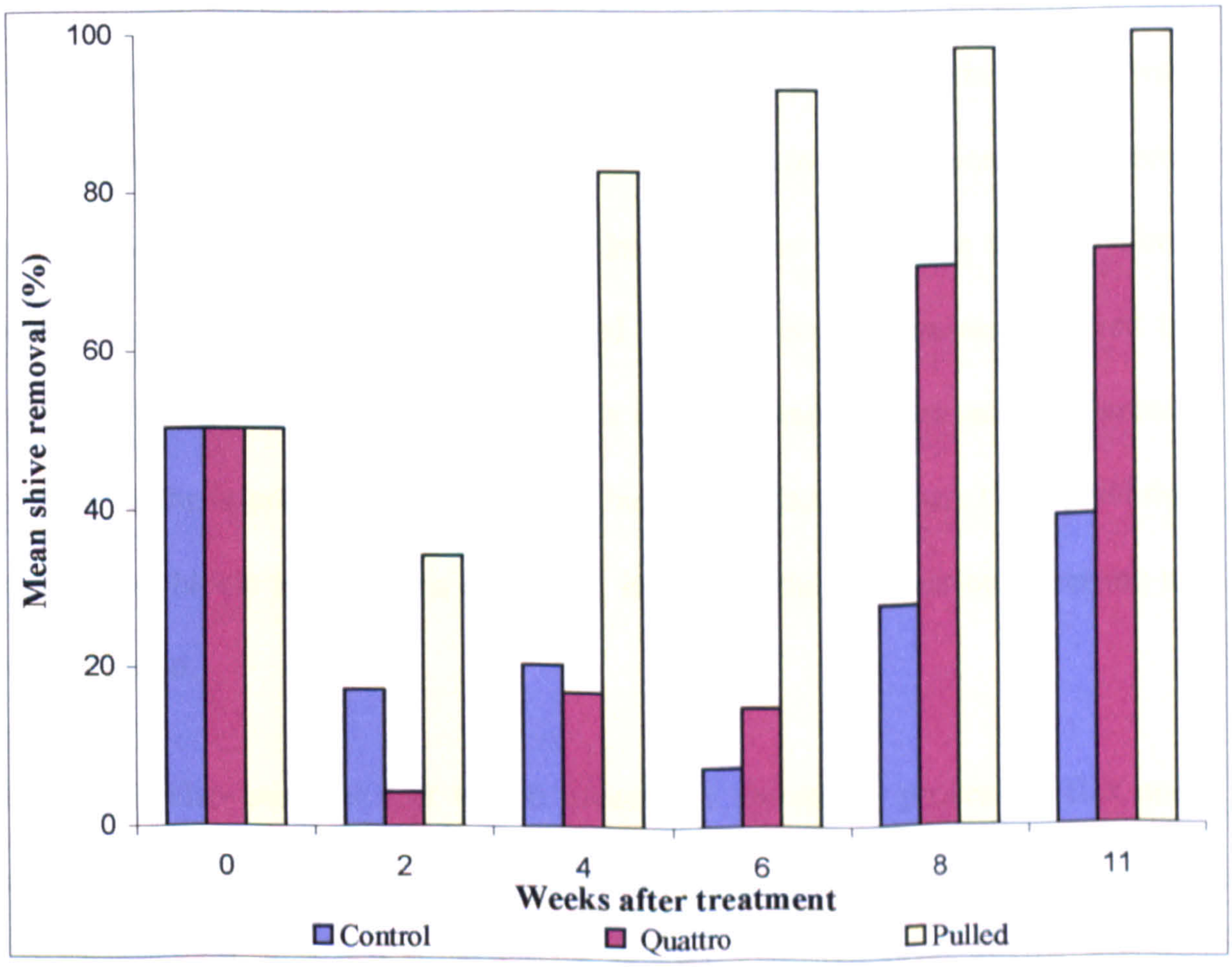


Fig. 25. Relative ease of decortication of flax straw after 2 decortication passes.

Chapter 5: Discussion

Retting is the most important stage in fibre production and it comprises two aspects, which develop concurrently:

a) the dissociation of the fibre bundles from the stem, and

b) the separation of the fibre bundles from each other and from the surrounding cortical tissues. There may also be a degree of sub-division of fibre bundles, which liberates individual ultimate fibres (single fibre cells).

Development of the peel and tear tests for use in monitoring retting

In the initial study to develop the technique used by Hampshire (1985), Goodman *et al.* (2002) showed that the work to peel decreased significantly during the retting period following desiccation of flax with glyphosate herbicides. It was concluded that this decrease in work to peel was due to colonisation of the stem, mostly by saprophytic fungi and that the largely pectic material in the inter-fibre matrix was digested by enzymes released by the micro-organisms involved in retting. The study indicated that peel tests could be used to measure mechanical changes at the interface between the fibre bundles (primary phloem tissue) and the secondary phloem tissue. Thus indirectly, it enabled the progression of retting to be monitored.

This study also used the tear test to investigate the retting process in flax and showed that the general shape of the curve for the tear force over the retting period was similar to that for the work to peel. However these results may be unreliable since the tear force was not related to the area of the fracture surface and the route taken by the fracture through the tissues is also rather unpredictable. It is likely that the fracture of cell walls, rather than the peeling apart of tissues,

occurred and this is supported by the general SEM evidence of cell debris attached to fibres (Plates 9 and 12). Nonetheless, it was concluded that the separation of fibre bundles from each other was affected by retting in a similar way and at a similar time as the dissociation of the fibre bundles from the woody core.

Subsequent investigations

These studies investigated two very different techniques; both of which enabled the decrease in work required to dissociate fibres from the stem during the retting period to be monitored and thus they may provide techniques that can be utilised to indirectly monitor the progress of the retting process itself in flax, hemp and other bast fibre crops. These techniques may lead to the development of equipment that can aid in-field management decisions for the improved timing of harvesting crops, or they may be of use to researchers for the evaluation of the effects of various parameters on the retting process, such as the effect of cultivar, plant density or stem diameter, species of retting micro-organisms, type of desiccant/dehydration used, maturity of plant and environmental conditions; the retting activity of commercially available enzymes may also be evaluated for use in industrial processing.

5.1 The effect of peeling angle on work to peel

As the sample being peel tested was rotated from the horizontally clamped position towards the almost vertically clamped position, the geometry of the peel test changed from that of a 90° peel test to that of the modified 180° peel test. The work to peel was significantly lower for the 90° peel test but as the sample was rotated, the work to peel began to increase when the sample was clamped at an

angle of 120° and became significantly higher when the angle of the peel test was 135°.

Thus, small changes in peeling angle had no significant effect on the work to peel ensuring that the error expected from inaccurate positioning of the sample in the jaws of the testing machine when large numbers of repeat tests are carried out had no significant effect on the results. However, data generated by the 90° peel test are not directly comparable with data from the modified 180° peel test.

It may be assumed that the slope of the flexible adherend (peel ribbon) at the point where it becomes detached from the rigid adherend (core of the stem) is zero (Williams, 1993). Indeed, as the test geometry was modified from a 90° peel test towards that of a 180° peel test, the slope of the peel ribbon at the point where it became detached from the core of the stem was observed to remain similar, while the curvature of the section of peeled tissue increased substantially. Thus, the work to peel remained almost constant at geometries close to the 90° peel test, but as the geometry of the test approached that of the 180° peel test, it is likely that the increased curvature of the section of peeled tissue dissipated increasing amounts of energy, and this may explain the greater work to peel values, as discussed by Williams (1993). The results from this study suggest that results from the two test geometries are not interchangeable and values for desiccated flax reported by Goodman *et al.* (2002) in a previous study using the modified 180° peel test cannot be compared directly with values for dew-retted hemp reported in this study using the 90° peel test.

There was no significant difference between the 90° peel test results for fresh hemp stems in the peeling angle and the dew retting investigations carried out in this study. The two sets of data produced work to peel values of 182 $\pm$ 13.0 J m^{-2}

and 186 ± 7.1 J m^{-2} respectively. The results from the 175° peel tests on fresh hemp stems in this study were very similar to the values found earlier by Goodman *et al.*, (2002) for the modified 180° peel test on flax stems; fresh hemp stems gave work to peel values of 241 ± 15.3 J m^{-2} while the comparable value for flax stems was 212 ± 7.9 J m^{-2}. If, as is suggested, the work to peel is largely determined by the composition of the intercellular adhesive that bonds the fibre bundles in place, it is likely that different species grown in different locations in different seasons would produce different peel test results. It is only to be expected that the composition of their adhesives would be determined by a complex interaction of genetic and site-specific environmental factors.

5.2 The effect of stem moisture content on work to peel

As stems dehydrated in the oven, the work to peel increased significantly (Fig. 12). This increase in work to peel is likely to be the result of changes in mechanical properties of both cell walls and inter-cellular materials; previous studies have shown that dehydration in a range of plant tissues (grass leaves, maize kernels, soybean pods) has increased stiffness and brittleness, affecting the characteristics of fracture (Vincent, 1983; Niklas, 1992). As moisture content decreases to around 20% work to fracture increases substantially, while at lower moisture contents there is increased variability in results (Fig. 14), perhaps indicating the onset of brittle fracture characteristics. Hepworth and Vincent (1998) performed tensile tests on xylem tissue conditioned at a range of humidities from 100% to 0% RH and found that dehydration increased the stiffness of xylem tissue by around 35%, with the greatest effect occurring between 100% and 60% RH.

In this study, the most dramatic increase in the work to peel occurred over a short period of time during which the moisture content decreased from 30% to 12%. This is also in agreement with other studies, which have reported particularly significant changes in mechanical properties of various plant tissues at moisture contents ranging between 50% and 20% and the onset of brittle fracture characteristics at around 13% moisture content (Vincent, 1990). Goodman *et al.* (2002) also reported that work to peel in flax stems increased to 540 J m^{-2} when the stems had dehydrated in the field to around 15% moisture content following the application of a desiccant herbicide.

In this study, peel tests conducted at mean stem moisture contents below 12% showed a dramatic decrease in work to peel (Fig. 12). This decrease, at very low moisture content, may be explained by the crack propagation characteristics of very dry materials. At these low moisture contents, the rapid decrease in the work to peel results from changes in stiffness that allow cracks to propagate more readily due to the development of brittle fracture characteristics (Vincent, 1990 & 1992). This effect could be readily observed during some of the peel tests on certain samples when the crack was propagated very rapidly and extended beyond the pre-set limits of the test method; the work to peel for such samples was very low. If industry could take advantage of this change to very low work requirement for decortication, then processing costs could be reduced considerably. However, commercially produced straw does not readily dry to less than 12% stem moisture content under field drying conditions and artificial drying on an industrial scale, to aid decortication, is unlikely to be economically viable. Moreover, the effects on fibre quality would have to be assessed and potential damage to fibres quantified.

5.3 The progress of retting in stand-retted flax crops

The results from this investigation indicate that the peel test can be used to indirectly monitor the progress of the retting process in stand-retted crops of flax. Two distinct phases in the retting process were identified (Fig. 15b). First, there was an increase in work to peel, which corresponded with the senescence and subsequent dehydration of the stem tissue due to the desiccant herbicide. Second, there was a decrease in the work to peel, (despite relatively constant moisture content), which corresponded with the progression of retting (Fig. 15b).

The dehydration of the stem tissue caused an increase in the work required to dissociate the peel from the woody core of the stem, either following desiccation with herbicide, or due to senescence (Fig. 15a). At first, the glyphosate-treated stems dehydrated more slowly than the untreated stems, so that after 5 days the moisture content of the treated stems had decreased by 5%, while in the untreated it had decreased by 10 %. The dehydration of the untreated stems may have been due to the onset of premature senescence caused by the very dry conditions during this study. Initially, the effects of drought stress were reduced by the application of the glyphosate herbicide. Indeed, observations during this period noted that the treated stems remained greener than the untreated stems. The initial effect of the herbicide may have been to induce loss of cell turgidity, closing the stomata, thus preventing moisture loss and temporarily reducing the effects of soil moisture deficit. Then, as the effects of the herbicide increased, the treated stems senesced and dehydrated more rapidly, reaching complete desiccation more quickly than the untreated stems (Fig. 15a).

Not surprisingly, the reduction in the moisture content of the stem corresponded with an increase in the work to peel. Studies by Niklas (1992) investigating the

effects of dehydration on plant tissues showed that the stiffness of plant tissue was indeed affected by its moisture content and that the Young's modulus (E) was increased in severely dehydrated tissues. Initially, before substantial dehydration of the tissues, there is also likely to be an effect caused by the loss of turgor pressure in cells (Vincent, 1990) as the senescing plants wilt.

The maximum value for work to peel was significantly greater for the treated stems than for the untreated stems (Fig. 15b), but in both cases it corresponded with the lower moisture of the stems at around 19 days after treatment. However, despite the moisture content of the stem remaining constant at around 10%, the work to peel decreased substantially, from 475 J m^{-2} to around 175 J m^{-2} at 54 days after treatment. This dramatic reduction in work to peel is likely to be due to the colonisation of the stem by the retting species. These micro-organisms release pectolytic enzymes, which digest the mostly pectic matrix binding the fibre bundles in position, allowing them to be separated relatively easily.

This study also shows that the work to peel decreased quicker in the treated stems than in the untreated stems (Fig. 15b). This is likely to have been due to the more rapid senescence of the stem tissues and hence earlier colonisation by retting species, but also could have been due to the direct action of the glyphosate in disrupting the cortical tissues (Fraser *et al.*, 1982). Indeed, Mercer and Fraser (1986) showed that in mature flax, glyphosate application disrupted the integrity of epidermal and cuticular tissues, this not only aids the dissociation of the fibre bundles, but also facilitates penetration of the stem tissues by fungal hyphae.

Variability in the results from the tear test may be due, at least in part, to variability in the fracture being monitored. Some peeling apart of tissues may well

have occurred, giving results similar to the peel test results, but there may well have been other aspects that need to be considered. Niklas (1992) suggests that lignified pectinaceous middle lamellae are stronger than the cell wall structure itself and that de-bonding of cells may be reduced. This suggests that in this test, the thin walls of the parenchyma cells between the bundles may be fractured, rather than the pectic material between the cells. This may have been increasingly likely as dehydration progressed and the thin cell walls became increasingly brittle. Further investigation of the fracture surface using scanning electron microscopy would help to clarify the route taken by the fracture. Also, if indeed the cells immediately adjacent to the fibre bundles do retain any characteristics of an endodermis and produce other non-polysaccharide materials such as suberin, the fracture between fibre cells and parenchyma cells will be further complicated.

The tissue peeled from the stems and used in the tear test bears some resemblance to the laminae of grasses in that there are discrete bundles of fibres running longitudinally, separated by thin walled parenchyma cells. Vincent (1983) showed that as the parenchyma cells between the fibre bundles of grass laminae dehydrated they became stiffer and their fracture characteristics changed; fracture between, and parallel to, the fibres became much easier. The fracture behaviour changed as the tissues dehydrated and these changes were explained at least in part by the decreasing amount of free water that was available to act as a plasticiser.

An increasing proportion of the water is bound to various cellular components of the structure making it unavailable to act as a lubricant between the various elements of the tissues. These changes in fracture behaviour are determined to a certain extent by the chemical composition of the tissues themselves, because the

nature and the quantity of the different chemical components will affect the availability of water. Cellulose and lignin will bind very little water, while pectins and hemicellulose will bind relatively more. In this study the situation is complicated further since both the quantity, and the relative proportions, of the components of the matrix between the fibres and the woody core of the stem change throughout the retting period as a result of the action of the retting enzymes.

In some cases, fracture may have occurred between fibre cells within the fibre bundles. This may also have been increasingly likely as the fibre bundles themselves began to dissociate as a result of the disintegration of the inter-fibre matrix. As dehydration and retting progressed and brittle fracture characteristics developed, cracks were propagated very rapidly and were often observed to extend beyond the test limits. It is important that future studies use techniques and test limits that will accommodate the extensive crack propagation that occurs following dehydration and subsequent retting of the stems.

This study indicates that the peel test provides a reliable technique for monitoring the changes in mechanical properties at the interface between the cellulose fibre bundles and the underlying stem tissues. It may be used to monitor the decrease in work to peel during the retting period and hence it can indirectly monitor the progress of pre-harvest retting in desiccated bast crops. It is particularly useful for monitoring the whole retting process including the earlier stages when relatively small affects cannot be evaluated using the traditional subjective methods. Although the peel test only monitors the dissociation of the fibre bundles from the woody core of the stem, this is likely to be the most important aspect of the retting process, in terms of work requirement.

The trouser tear test may be used to indicate the progress of retting by monitoring the ease with which fibre bundles can be separated from each other and from the surrounding cortical tissues. However, the actual process being monitored must be further clarified in order to enable effective interpretation of the data produced. Due to the variability of the results, it is unlikely that the tear test will be of practical use in monitoring retting of bast crops, without substantial further development.

5.4 The progress of retting in pulled and dew-retted hemp

The dew-retted stems dehydrated relatively slowly in the field, taking around 28 days to reach 12% moisture content (Fig.18b) and during this time the stems were exposed to microbial colonisation and the associated retting process. The significant increase in work to peel observed in the oven-dried stems was not evident in the dew-retted stems (Figs 18a and 19). It is likely that the marked reduction in work to peel of the dew-retted hemp stems was due to the disintegration of the inter-cellular matrix during the retting process.

This study shows that peel tests can be used to objectively monitor the reduction in work to peel fibre bundles from the core of the stem in hemp. This reduction in work to peel indicates the progress of retting and thus the peel test can be used to indirectly monitor the progress of retting in dew-retted hemp crops. As with flax, in future work this technique may be used to investigate the relationship between fungal colonisation and changes in the mechanical properties of the stem tissue and evaluate the effects of different agronomic factors on the ease of decortication in hemp.

5.5 The ease of decortication of enzyme-retted flax following increasing periods of time in a retting solution.

From direct observations it was clear that some of the shive that had been detached from the fibre, despite being loose, remained entangled within the mesh of fibre ribbons. This entanglement of loose shive was variable and inconsistent; hence the relationship between the length of time spent in the retting solution and the subsequent ease of separation of fibre bundles from the stem was masked. The results obtained from the initial experiment showed no relationship between the duration of the retting period and the ease of decortication as measured by the weight of shive removed during the series of decortication passes.

During decortication, the fibre layer tended to be detached from the core of the stem in continuous ribbons, and a proportion of stem debris proved difficult to remove effectively from this mat of ribbons. It was apparent that loose but entangled debris was being included in the weights, making results inaccurate. The extra energy used to disentangle this trapped debris was difficult to administer consistently and virtually impossible to accommodate in the subsequent interpretation of results.

A method of removing this debris with the minimum extra effect on the decortication of the sample was devised. A low velocity airstream from a vacuum pump was used to remove the debris effectively. In order to retain the sample and prevent loss of fibre, the ribbons were firmly gripped and the airstream applied in a longitudinal direction; each sample was held in the airstream for 3 seconds after each decortication pass before being re-weighed. In order to confirm that fibres were not also being extracted by this secondary process, pre-cleaned samples were subjected to repeated decortication passes and exposed to the airstream shive

extraction system, weights after several additional decortication passes showed no further loss of weight indicating that fibres were not being removed in the airstream. This modification improved the reliability of the data substantially.

The inclusion of loose but entangled shive and debris in the weight of the fibre after decortication ensured that the results from the initial experiment produced no useful data in terms of the ease of decortication of enzyme-retted straw (Fig. 20a). However, the modified method's use of a low velocity airstream to remove the loose shive and debris from the sample, without administering any additional mechanical work, produced much more reliable and interesting data (Fig. 120b). These results showed that the longer the sample was left in the retting solution (the longer the duration of the retting period) the more effective the decortication. Not only was a greater proportion of shive removed, but it was removed by fewer passes through the decortication system. This is likely to be due to the polysaccharide complex that bonds the fibre bundles in place being digested by the enzymes present in the retting solution.

As the exposure of the sample to the activity of the enzymes is extended, the integrity of the cement bonding the fibre bundles to the woody core of the stem is disrupted and it becomes easier to dissociate the fibre bundles from the stem structure. This reduction in the energy to separate the fibre bundles from the stem was clearly shown by the more effective decortication.

Thus, the inclined plane decorticator provides a useful technique for monitoring the disintegration of the structure that binds the fibre bundles to the woody core of the flax stem and it is now in regular use for monitoring the progress of retting in flax by the TEAM research group at De Montfort University. Similarly, following

a demonstration, the inclined plane decorticator has also been adopted by researchers at University of Wales, Bangor to assess the effects of various parameters on the progress of retting in flax.

5.6 The ease of decortication of stand-retted flax crops

Generally, stem decortication initially became less effective beyond the mid-point of flowering (MPF). This initial effect was greater in herbicide-treated stems, but less in pulled stems, than in the untreated controls (Fig. 25).

Decortication of untreated control stems was less effective as the plants matured beyond flowering than it was at MPF. This must have been due to the changing nature of the materials at the fracture surface between the fibres and the core of the stem as the plant matured. Many changes occur within plants at this time, but increasing lignification could be one of the more important factors responsible for this effect. Eventually, the untreated stems again became easier to decorticate, perhaps as the stems naturally senesced and became colonised by fungi and the retting process began, but decortication was never as effective as it was at MPF.

Similar effects were observed in the treated stems, but as the effect appears to be greater than in the control stems, there may be other additional factors at work here. There may be direct chemical changes induced by the herbicide or alternatively chemical changes induced by the stress of reduced availability of soil moisture. Glyphosate-treated stems senesce relatively slowly and may continue to survive and grow under conditions of great stress for some time. On the other hand, the pulled plants senesce much more rapidly and are subjected to a relatively short period of stress.

The untreated plants continue to grow normally and their lignin contents may increase rapidly during the period immediately following MPF. Herbicide treated plants may retain such an ability to lignify tissues for a short period after application of the desiccant herbicide, indeed the process may be encouraged in response to stress. Pulled plants are unlikely to be able to lignify tissues further due to the rapid nature of their senescence and any response to stress would be small.

Sample preparation may have complicated interpretation of the results. The rapid drying at relatively high temperatures may have influenced the nature of chemicals such as lignin. The untreated controls were dried from fresh (relatively high moisture contents), while desiccated stems initially dried more slowly (due to the action of the herbicide treatment). Thus, desiccated stems were relatively dry before the high temperature drying process was implemented and any effects on materials such as lignin may have been different for the untreated controls than for the desiccated stems. The difference in moisture content between the control stems and the treated stems was not constant, it increased over the 4 weeks immediately following treatment as the treated stems dehydrated, but then remained relatively constant thereafter.

If stems were effectively dehydrated prior to the end of flowering, decortication became more effective over time and this is thought to be due to the retting process; colonising fungi secrete enzymes, which digest the matrix material cementing the fibres in position and facilitate mechanical separation. Untreated stems did not dehydrate or decorticate effectively. More than 10 weeks beyond MPF these stems still retained 55% moisture content and required considerable

decortication work to separate the fibres from the shive, a series of 4 decortication passes only removed around 75% of the shive (Figs 23 and 25).

In this study, desiccant herbicides were only partially successful in desiccating the crop. Other investigations have previously highlighted similar problems and it may be a wise precaution to use more robust application rates to reduce the risk of ineffective desiccation in future investigations.

The application of Quattro provided quite effective chemical desiccation, but only reduced moisture content to around 20% after 4 weeks. The causes of ineffective desiccation are not fully understood, but plant maturity at application, and possibly weather conditions (over a period of several days around application), were likely to have been contributing factors.

After a period of retting, decortication became significantly more effective and after 8 weeks 2 decortication passes removed around 70% of the shive. However, increasing the decortication work to 4 decortication passes only enabled up to 90% of shive to be removed. This may indicate that retting was incomplete and this in turn may be a result of the limited desiccation achieved; the pulled plants, which dehydrated very quickly reached similar levels of decortication after only 4 weeks. A prolonged retting period may have improved results for the herbicide desiccated stems, but this was impractical due to agronomic requirements.

In this season, pulling and stand-retting produced stems that decorticated most effectively. Dehydration of the stems was very effective; moisture content of the pulled stems decreased to below 15% within 7 days and retting progressed effectively; after 8 weeks a single decortication pass gave complete decortication.

The first decortication pass does little more than flatten the sample, unless the sample is very well retted, it is the second pass that is more effective in decorticating the sample and differentiating between treatments. However, the subsequent decortication passes are necessary in order to achieve acceptable levels of decortication, but the result for each successive pass is dependent on the efficacy of the previous pass(es) and so it is the second pass that provides the best evidence for the ease of decortication assessment (Fig. 25). The data from the second decortication pass shows the substantial differences between the treatments (Fig. 24). Although all the treatments become more effectively decorticated over time, the period of time required seems to be related to the dehydration of the stems. The control stems did not decorticate effectively within the duration of the study, the desiccated stems decorticated relatively effectively after around 8 weeks, and the pulled stems decorticated very effectively after only 4 weeks. These differences are likely to be due to the differences in colonisation of the stems by retting species. The untreated control stems remain alive and are not significantly colonised until much later, when natural senescence begins. Colonisation is relatively slow in the desiccated stems and relatively fast in the pulled stems, and this may be related to the moisture content or senescence of the stems, stem dehydration to around 15% moisture content takes about 4 weeks in desiccated stems, but only about 7 days in pulled plants (Fig. 21).

The design of the equipment used in this investigation is suitable for the decortication of flax, but it was not possible to decorticate hemp stems using the same design. The profile of the belt and roller was too shallow; the roller was lifted off the belt and "derailed" as it passed over even the most slender hemp

stems. A deeper profile for both the belt and the roller and indeed a heavier roller would be required in order to successfully transfer this technique to hemp.

However, an alternative technique may be considered: longitudinal strips cut from the flatter areas of the stem, may be suitable for decortication in this way, although longitudinal cut stems may tend to splay or curve due to pre-tension in the outer layers of the stem tissues.

Conclusions

Only part of the work done while investigating fibre-crops at De Montfort University is reported here; i.e. those aspects relating to the development and evaluation of novel test methods for investigating the retting and decortication of bast fibres.

The progression of retting in flax and hemp can be monitored indirectly by employing standard mechanical tests such as the peel test, tear test and ease of decortication test (inclined plane decortication). This study developed test methods and showed that retting could be monitored using these techniques, in enzyme-retted flax, stand-retted flax and dew-retted hemp straw.

As the complex inter-cellular matrix that cements the fibres into position within the stem is digested by the activity of microbial enzymes, the work done to dissociate the fibres from the stem decreases. Peel tests can be employed to measure this decreasing work to peel and thus the progress of retting can be monitored. The geometry of the peel test is important in so much as the results from the 90° test are substantially lower than for the modified 180° test. However, comparative tests that are carried out at the same angle are valid.

The relative ease of decortication of flax stems can be monitored directly using the inclined plane decorticator developed in this study. As retting progresses, the adhesion of fibres to the other stem tissues becomes less robust and they are more easily decorticated between the profiled roller and the belt. The proportion of shive removed increases and the work required to dissociate the fibres decreases as the length of the retting period is extended..

The tear test can measure the work done in separating fibre bundles from each other and the surrounding cortical tissues, indicating the progress of retting. However, due to a less uniform fracture, the data produced is more variable and less reliable than the peel test data. It is unlikely that the tear test will be of any great value in monitoring the progress of retting without substantial further development.

In this study peel tests enabled comparisons of the retting characteristics of stand-retted flax straw to be investigated and compared. Stems that had been stand-retted following desiccation with glyphosate were compared with untreated stems. The test illustrated significant differences between desiccated and untreated stems, even when the untreated stems senesced naturally due to drought stress.

The peel test also identified changes in the fracture characteristics of hemp stems that were unrelated to retting. When stems were dried in a laboratory oven at 40° C, the increasing stiffness of the stems due to dehydration and changes in the fracture characteristics as stem moisture content decreased were identified.

The ease of decortication test developed in this study was used to investigate the activity of a standard enzyme-retting solution on flax straw. The test proved sensitive enough to identify significant differences in ease of decortication of samples that had been exposed to differing durations of retting period. The longer the retting period, the greater the proportion of shive removed and the fewer decortication passes required to decorticate the sample.

The ease of decortication in stand-retted flax straw was also investigated; glyphosate desiccated straw was compared with untreated straw and pulled straw after stand-retting for up to 11 weeks. The test identified significant differences

between the treatments, the longer the duration of retting the easier the decortication. Desiccation with glyphosate may interfere with the retting process, although the mechanism for this is not yet understood, it is unlikely that moisture content alone is responsible for the observed effects. The test also identified that in untreated plants, decortication became significantly more difficult after the plants had begun to flower. Again the mechanism for this is not yet understood, but it could have important implications for the timing of pulling flax crops in the traditional pulled and dew-retted production system.

The inclined plane decorticator proved to be unsuitable for hemp due to morphological features of the hemp stem. The profile of the timing belt and the timing gear were too shallow to accommodate the more robust hemp stems. However, with modification the ease of decortication test may prove to be as effective in hemp as it is in flax.

Further Work

Peel test

The cyclical nature of the peel tests (Fig. 3) may be used to investigate the changing nature of the fracture characteristics as the stems senesce, dehydrate and ret. The peeling action may be considered simply as two processes; bending of the peel and then separation of peel from the core of the stem by breaking bonds. Initially, the force applied is not sufficient to break bonds and detach the fibres from the core of the stem and the peel will be deformed, the applied force increases over time until it is sufficient to separate the fibres from the core. This cycle is repeated for the next portion of the peel. Thus the frequency and amplitude of the trace will be affected by the fracture characteristics and may be used to investigate the fracture at different moisture contents or stages of retting. Effective investigation of the frequency and amplitude of the force versus displacement traces will require the collection of substantially larger data sets.

There appears to be an increase in variability of data at certain moisture contents that may indicate a change in the fracture characteristics. This could be associated with the senescence of the tissues and loss of turgor in cells, but it may also be influenced by a change in the nature of the intercellular cement that binds the fibres in position. The physical and chemical characteristics of this intercellular layer (and their interactions) should also be considered. The peel test data exhibits increased variability between 40% and 60%, at around 20% and again below 10% moisture content. Further investigations should concentrate in these areas particularly.

The SEM evidence observed in this work proved to be inconclusive and further observations should be made to clarify the route of the fracture and describe the fracture surface. In conjunction with other work described above this should inform a better understanding of the separation of the fibres from the core of the stem.

The peel test may be used to investigate the effects of a wide range of parameters on the progress of retting, such as cultivar selection, plant density/stem diameter, or retting species.

Tear Test

The fracture characteristics in the tear test should be investigated to clarify the suitability of the tear test in monitoring retting. The adhesion and subsequent separation of contaminating cortical tissues from the fibre cells is of great interest to both textile and composite end users. The actual route of fracture should be determined to establish whether cell walls are fractured or whether tissues are being peeled apart. The fracture surface should be described using SEM. In order to enable the force required to tear the peel strip to be related to the area of the actual fracture surface.

Inclined Plane Decorticator

The inclined plane decorticator does not measure the work done to dissociate the fibres, but a method for including such a measurement would be very beneficial. This could be achieved by considering the change in kinetic energy of the roller as it decorticates the stem, indicating the decortication work. A motion sensor fitted to the upper end of the inclined plane could monitor the velocity of the roller. The kinetic energy of the roller could be calculated by rolling it down the inclined

plane, in the absence of any straws. The decortication energy can be considered as being equal to the roller's loss of kinetic energy as it passes over the straw. Comparative data could be easily produced, but the area of the fracture surface would need to be calculated in order to produce reliable absolute values for decortication work. Kinetic energy converted to other energy forms such as sound and heat would also need to be considered.

References

Akin DE, Gamble GR, Morrison WII, Rigsby LL and Dodd R B. 1996. Chemical and structural analysis of fibre and core tissues from flax. *Journal of the Science of Food and Agriculture* 72:155-165.

Akin DE, Morrison WII, Gamble GR, Rigsby LL, Henriksson G, and Eriksson KL, 1997. Effect of retting enzymes on the structure and composition of flax cell walls. *Textile Research Journal* 67:279-287.

Akin DE, Morrison WII, Rigsby LL and Dodds RB. 2001. Plant factors influencing enzyme retting of fiber and seed flax. *Journal of Agriculture and Food Chemistry* 49:5778-5784.

Archibald DD, Henrikssen G, Akin DE, and Barton FE. 1998. Spectroscopic characterisation of enzymatic flax retting: factor analysis of FT-IR and FT Raman data. *American Institute of Physics Conference Proceedings* 403:653-656.

Bayliss AD, Hart CA, Howell A, Townson JK and Ngim J. 1996. Sulfosate: A new non-selective herbicide for broad-spectrum weed control. *Proceedings of MAPPS/ISP seminar 'A new era in weed management'*. Zeneca Agrochemicals - Asia Herbicide Development Conference.

Berk RA. 2003. *Regression analysis: A constructive critique*. Thousand Oaks, CA: Sage Publications.

Bratt RP, Mercer PC, Brown AE. 1988. Degradation of flax stems by *Botrytis cinerea*. *Transactions of the British Mycological Society* 90:537-544.

Brown AE. 1984. *Epicoccum nigrum*, a primary saprophyte involved in the retting of flax. *Transactions of the British Mycological Society* **83**:29-35.

Brown AE and Sharma HSS. 1984. Production of polysaccharide-degrading enzymes by saprophytic fungi from glyphosate-treated flax and their involvement in retting. *Annals of Applied Biology* **105**:64-74

BS EN 28510-1:1993. Adhesives – Peel test for a flexible-bonded-to-rigid test specimen assembly – Part 1: 90° peel. ISBN 0 580 19629 1.

BS EN 28510-2:1993. Adhesives – Peel test for a flexible-bonded-to-rigid test specimen assembly – Part 2: 180° peel. ISBN 0 580 19630 5.

Courtney AD and Robinson E. 1982. Glyphosate (N-(Phosphonomethyl) glycine) as a pre-harvest retting agent in flax (*Linum usitatissimum*). *Proceedings of the 1982 British Crop Protection Conference: Weeds (Brighton)*, British Crop Protection Council, Croydon, UK. pp.231-236.

Eames AJ and MacDaniels LH. 1947. *An Introduction to Plant Anatomy.* New York: McGraw-Hill.

Easson DL and Cooper K. 2002. A study of the use of the trimesium salt of glyphosate to desiccate and ret flax and linseed (*Linum usitatissimum)* and of its effects on yield of straw, seed and fibre. *The Journal of Agricultural Science* **138**:29-37.

Easson DL and Long NJ. 1992. Pre-harvest retting of flax with glyphosate. In *The Biology and Processing of Flax*, pp. 213-228. Eds H S S Sharma and C F Van Sumere. Belfast: M Publications.

Easson DL and Molloy R. 1996. Retting – A key process in the production of high value fibre from flax. *Outlook on Agriculture* **25:235-242.**

East Midlands Development Agency. 2002. Lincoln hemp trial. A one year development project.

Esau K. 1965. *Plant Anatomy* (second edition.). New York: Wiley.

Esau K. 1977. *Anatomy of Seed plants.* New York: Wiley.

Fahn A. 1990. *Plant Anatomy* (fourth edition.). Oxford: Pergammon Press.

FAIR CT-98 9574. 1998. Cultivation and processing systems for the cost efficient recovery of fibres from flax for use as a staple fibre. A 2 year EC sponsored project under the FAIR programme.

Feng PCC, Ryserse JS & Sammons RD. 1998. Correlation of leaf damage with uptake and translocation of glyphosate in velvet leaf. *Weed Technology* **12**: 300-307.

Fibrenova Ltd. 2001. A comparison of several hemp cultivars grown in UK conditions. A one year industry sponsored project.

Fila G, Manici LM and Caputo F. 2001. In vitro evaluation of dew-retting of flax by fungi from southern Europe. *Annals of Applied Biology* **138:343-351.**

Fraser TW, Courtney AD and Harvey BMR. 1982. Pre-harvest retting of flax – a light microscope study of the effects of glyphosate treatment on the maturation of stem tissues. *Annals of Applied Biology* **101:533-537.**

Goodman AM, Ennos AR and Booth I. 2002. A mechanical study of retting in glyphosate treated flax stems (*Linum usitatissimum*). *Industrial Crops and Products* **15**:169-177.

Gorshkova. TA, Wyatt SE, Salnikov VV, Gibeaut DM, Ibrgimov MR, Lozovaya VV, and Carpita NC. 1996. Cell-wall polysaccharides of developing flax plants. *Plant Physiology* **110**:721- 729.

Gubbels GH and Kenaschuk EO. 1981. Desiccation of flax with diquat. *Canadian Journal of Plant Science* **61**:575-581.**Hampshire RJ. 1985.** A study in the social and reproductive behaviour of captive grey squirrels (*Sciurus carolinensis*). PhD Thesis, University of Reading.

Harvey BMR and Crothers SH. 1988. Causes of uneven desiccation of flax by glyphosate: poor penetration of the canopy and variable spray deposition. *Annals of Applied Biology* **112**:195-200.

Harvey BMR, Spence L and Crothers SH. 1985. Pre-harvest retting of flax with glyphosate: effect of growth stage at application on uptake, translocation and efficacy of glyphosate. *Annals of Applied Biology* **107**:263-270.

Hatfield RD, Ralph J, and Grabber JH. 1999. Cell wall crosslinking by ferulates and diferulates in grasses. *Journal of the Science of Food and Agriculture* 79:403-407.

Hepworth DG and Vincent JFV. 1998. The Mechanical Properties of Xylem Tissue from Tobacco Plants (*Nicotiana tabacum* `Samsun'). *Annals of Botany* **81**: 751-759.

His I, Marry M, Huxham IM, Tetley L and Jarvis MC. 2001, in press. EELS localisation of boron in flax fibres. In *Boron analysis and imaging in biological materials*. Ed. M. Thellier.

Hobson J. 2001. Personal communication. Hemcore Ltd, Bishop Stortford, UK.

Hock CW. 1942. Microscopic structure of flax and related bast fibres. *Journal of Research for the National Bureau of Standards* **29**:41-50.

Houtman CJ, Atalla RH. 1995. Cellulose-lignin interactions. *Plant Physiology* **107**:977-984.

Iiyama K, Larn TBT and Stone BA. 1990. Phenolic acid bridges between polysaccharides and lignin in wheat internodes. *Phytochemistry* **29**:733-737.

Iivvessalo-Pfaffii MS. 1995. *Fiber atlas identification of papermaking fibers.* Berlin: Springer.

Jarvis MC, Briggs SPH and Knox JP. 2003. Intercellular adhesion and cell separation in plants. *Plant, Cell & Environment* **26**: 977-989.

Jauneau A, Cabin-Flaman A, Verdus MC, Ripoll C and Thellier M. 1994. Involvement of calcum in the inhibition of endopolygalacturonase activity in epidermis cell walls of *L. usitatissimum. Plant Physiology* **32**:839-846.

Jung HG, Ni W, Chapple CCS and Meyer K. 1999. Impact of lignin composition on cell wall degradability in an *Arabidopsis* mutant. *Journal of the Science of Food and Agriculture* **79**:922-928.

Karus M and Leson G. 1995. Textiles from hemp fibers – new ways for German hemp. *Journal of the International Hemp Association* **2**:101-102.

Keller A, Leupin M, Mediavilla V and Wintermantel E. 2001. Influence of the growth stage of industrial hemp on chemical and physical properties of the fibres. *Industrial Crops and Products* **13**:35-48.

Kessler RW, Becker U, Kohler R and Goth B. 1998. Steam explosion of flax – a superior technique for upgrading fibre value. *Biomass and Bioenergy* **14 (3)**: 237-249.

Kirby RH. 1963. *Vegetable Fibres: Botany, cultivation and utilization.* New York: Interscience Publications Incorporated. pp.15-37.

Kohler R, Kessler RW. 1999. Designing natural fibres for advanced materials. *Proceedings of the 5th International Conference on Woodfiber-Plastic Composites, Madison, USA.*

Kowslowski R, Rawluk M and Barriga J. 2002. Future directions in usage of natural renewable raw materials. *Proceedings of the Production, Processing and Use of Natural Fibres Conference, Potsdam.*

Lewis GN, Davin LB and Sarkenen S. 1996. Lignin and Lignan Biosynthesis: Distinctions and Reconciliations, *American Chemical Society Symposium Series No 697*, ch 1, pp 1-28.

Lewis NG. 1999. A 20th century roller coaster ride: a short account of lignification. *Current Opinions in Plant Biology* **2**:153-162.

Love GD. 1994. Determination of phenolic structure in flax fibre by solid state CNMR. *Phytochemistry* **35**:489-491.

Meijer WJM, Vertregt N, Rutgers B and van der Waart. 1995. The pectin content as a measure of the retting and rettability of flax. *Industrial Crops and Products* **4**:273-284.

Mercer PC and Fraser TW. 1986. Micro-organisms associated with the retting of flax treated with the herbicide glyphosate. *Annals of Applied Biology* **109**:509-521.

Morrison WH, Himmelsbach DS, Akin DE, and Evans JD. 2003. Chemicals and spectroscopic analysis of lignin in isolated flax fibres. *Journal of Agricultural and Food Chemistry* **51**:2565-2568.

Morvan C, Andeme-Onzighi C, Girault R, Himmelsbach DS, Driouich A, and Akin DE. 2003. Building flax fibres: more than one brick in the walls. *Plant Physiology and Biochemistry* **41**:935-944.

Naldrett MJ. 1992. Adhesives. In *Biomechanics – Materials: A practical approach*, pp. 230-231. Ed JFV Vincent. Oxford: Oxford University Press.

Niklas KJ. 1992. Voigt and Reuss models for predicting changes in Young's modulus of dehydrating plant organs. *Annals of Botany* **70**:347-355.

Niklas KJ and O'Rourke TD.1987. Flexural rigidity of chive and its response to water potential. *American Journal of Botany* **74**:1033-1044.

Rudkin S. 2001. Personal communication. Industrial Crop Partnership Ltd, Leicester, UK.

Schenek A. 2002. Natural fibres – production and importance. *International Textile Bulletin* **2**:8-17.

Schroeder D, Sjoquist DL, and Stephan PE. 1986. *Understanding regression analysis: An introductory guide*. Thousand Oaks, CA: Sage Publications. Series: Quantitative Applications in the Social Sciences, No. 57.

Seaby DA and Mercer PC. 1984. Development of a hand tool to test the degree of retting of flax straw. *Annals of Applied Biology* **104**:567-573.

Sharma HSS. 1986a. Effect of glyphosate treatment on lignification of fibres of some flax cultivars. *Tests of Agrochemicals and Cultivars* 7:114-115. A supplement to *Annals of Applied Biology*.

Sharma HSS. 1986b. An alternative method of flax retting during dry weather. *Annals of Applied Biology* **109**:605-611.

Sharma HSS. 1988. Chemical retting of flax using chelating compounds. *Annals of Applied Biology* **113**:159-165.

Sharma HSS and Faughey GJ. 1999. Comparison of subjective and objective methods used to assess flax straw cultivars and fibre quality after dew-retting. *Annals of Applied Biology* **135**:495-501.

Sharma HSS and Van Sumere CF. 1992. *The Biology and Processing of Flax.* Belfast: M Publications.

Sharma HSS, Lefevre J and Boucaud J. 1992. Role of microbial enzymes during retting and their effect on fibre characteristics. In *The Biology and Processing of Flax*, pp. 199-212. Eds HSS Sharma and CF Van Sumere. Belfast: M Publications.

Sharma HSS, Mercer PC and Brown AE. 1989. A review of recent research on the retting of flax in Northern Ireland. *International Biodeterioration* **25**:327-342.

Sprankle P, Meggitt WF and Penner D. 1975. Absorption, action and translocation of glyphosate. *Weed Science* **23**:235-240.

Sustainable Technologies Initiative. 2004. *Urtica dioica* - A Novel Sustainable Fibre Crop for the UK.

Talmadge KW, Keegstra K, Bauer WD and Albersheim P. 1973. The structure of plant cell walls. I. The macromolecular components of the walls of suspension-cultured sycamore cells with a detailed analysis of the pectic polysaccharides. *Plant Physiology* **5**1:158- 173.

TEXFLAX. 2002. The processing and cultivation of short fibre flax for high value textile end uses. LINK project no. F24 DeMont03RH.

Tiver NS. 1942. Physiology of growth, stem anatomy and fibre development in fibre flax. *Australian Journal of Experimental Biology and Medical Science* **20**:149-160.

Turner AJ. 1949. The structure of flax. *Journal of the Textile Institute* **40**:1-12.

Van Sumere CF. 1992. Retting of flax with special reference to enzyme-retting. In *The Biology and Processing of Flax*, pp. 157-198. Eds HSS Sharma and CF Van Sumere. Belfast: M Publications.

Van Sumere CF and Sharma HSS. 1991. Analyses of fine flax fibre produced by enzymatic retting. *Aspects of Applied Biology* **28**:15-20.

Vincent JFV. 1983. The influence of water content on the stiffness and fracture properties of grass leaves. *Grass and Forage Science* **38**:107-114.

Vincent JFV. 1990. Fracture properties of plants. *Advances in Botanical Research* **17**:234-287.

Vincent JFV. 1992. Fracture. In *Biomechanics – Materials: A practical approach*, pp.193-217. Ed JFV Vincent. Oxford: Oxford University Press.

Wang HM, Postle R, Kessler RW and Kessler W. 2003. Removing pectin and lignin during processing of hemp for textile applications. *Textile Research Journal* **73**:664-669.

Whitford AC. 1947. Bast Fibres. In *Matthews' Textile* Fibers (5th edition). flax pp. 305-325, hemp pp. 337-345. Ed HR Mauersburger. New York:Wiley.

Williams JG. 1993. Root rotation and plastic work effects in the peel test. *Journal of Adhesion* **41**:225-239.

Publications

Peer-reviewed Journals

Booth I, Goodman AM, Grishanov SA & Harwood RJ. 2004. A mechanical investigation of the retting process in dew-retted hemp (*Cannabis sativa*). *Annals of Applied Biology* **145** (1): pp. 51-58.

Booth I, Harwood RJ, Wyatt JL & Grishanov SA. 2004. A comparative study of the characteristics of fibre-flax (*Linum usitatissimum*). *Industrial Crops and Products* **20**: pp. 89-95. A Special Edition from the GreenTech Conference (2002).

Goodman AM, Ennos AR and Booth I. 2002. A mechanical study of retting in glyphosate treated flax stems (*Linum usitatissimum).* *Industrial Crops and Products* **15**: 169-177.

Conference Proceedings

Booth I & Goodman AM. 2003. A mechanical study of retting in flax (*Linum usitatissimum L.)* and hemp (*Cannabis sativa L.)* stems. *Comparative Biochemistry and Physiology* **134**: March supplement pS45.

Goodman AM and Booth I. 2000. A mechanical study of retting in glyphosate treated flax stems (*Linum usitatissimum L.). Journal of Experimental Botany* **51**: March supplement p48.

Appendices

Appendix 1 - Test geometry for the 90° peel test

Crosshead height at start of test = h
Displacement = D
Peel length = P
Crosshead height after test = H (Where h + D = H)

Then according to Pythagorus:

$H^2 + P^2 = (h + P)^2$

$H^2 + P^2 = h^2 + 2hP + P^2$

$H^2 = h^2 + 2hP$

$P = (H^2 - h^2)/2h$

Specific example calculation for a extension assembly of 650 mm:

1. If h = 650 mm and displacement D = 10 mm
   Then H = 660 cm

   The length of peel $P = (660^2 - 650^2)/(2 \times 650)$
   $= (435600 - 422500)/ 1300$
   $= 13100/1300$
   $= 10.077mm$
   The assumption that p = 10mm is incorrect.
   However, the error is negligible:

   $(0.077/10) \times 100 = 0.77$ % error

   This is the maximum error that would occur.

2. The angle of deviation of the direction of load from the vertical is 0.87 degrees.

3. The length of peel required to give a significant error is when angle (A) = 6 °:

   $\sin A = P/h = 0.1045$
   $P = 0.1045 h + 0.1045P$
   $P - 0.1045P = 0.1045h$
   $P(1 - 0.1045) = 0.1045h$
   $0.8955 P = 0.1045 h$
   $P = 0.1045h/0.8955$

   $P = (0.1045 \times 650)/0.8955 = 75.87mm$

Appendix 2 - Test geometry for peel test at angle 0°

Crosshead height at start of test = h
Displacement = D
Crosshead height after test = H. (Where h + D = H)

BUT effective height of crosshead = Z = H-a
Where a = height of peel front above horizontal at end of peel test

Peel length along inclined stem = P
P forms the diagonal of the rectangle a x b
Where b = horizontal displacement from vertical start of test.
(See test geometry diagram)

So: $a = P \sin\theta^\circ$
$Z = H - a = H - (P \sin\theta^\circ)$
$b = P \cos\theta^\circ$

Thus,

$$(h + P)^2 = b^2 + Z^2$$
$$h^2 + 2hP + P^2 = (P \cos\theta^\circ)^2 + (H - P \sin\theta^\circ)^2$$
$$= P^2 \cos^2\theta^\circ + H^2 - 2H P \sin\theta^\circ + P^2 \sin^2\theta^\circ$$
$$= P^2 (\cos^2\theta^\circ + \sin^2\theta^\circ) + H^2 - 2H P \sin\theta^\circ$$

Since $(\cos^2\theta^\circ + \sin^2\theta^\circ) = 1$, and the P^2 cancel out:

$$h^2 + 2hP = H^2 - 2H P \sin\theta^\circ$$
$$2 h P = H^2 - 2H P \sin\theta^\circ - h$$
$$2 h P + 2H P \sin\theta^\circ = H^2 - h^2$$
$$P (2h + 2H \sin\theta^\circ) = H^2 - h^2$$
$$P = (H^2 - h^2)/(2h + 2H \sin\theta^\circ)$$

If $\theta^\circ = 0$, then $\sin\theta^\circ = 0$, ie for horizontal sample position and:

$$P = (H^2 - h^2)/2h.$$

This is the maximum error for P (0.77 %) as any increase in angle will add a value for $\sin\theta^\circ$ to the denominator and thus reduce the value for P until angle = 90°, at which point P = 10 mm.

The extension piece causes a slight problem as some downward force is applied from its centre of gravity, out of line with the direction of the peel test, but again this is negligible since the angle from vertical is negligible.

As the angle of peel changes, the peel force becomes inaccurate, since it is related to $\cos\theta^\circ$. The crucial angle is 6° because $\cos 6^\circ = 0.99$ and would give an error of 1 %. At a peel angle of 6° and using a 65cm extension piece, the peel length would be 75mm on a horizontally clamped specimen.

Appendix 3 - Estimation of work to tear based on Goodman *et al.*, 2002.

Mean thickness of peel	=	0.125 mm
Length of peel	=	10.00 mm
Surface area of fracture	=	1.25 mm^2
Mean tear force	=	0.0272 N
Mean work to tear	=	(0.0272/1.25) x 10,000
	=	218 J m^{-2}

Approximation only.

Assumptions:

Peel thickness is uniform along the length of the peel section.
Peel thickness is uniform along width of peel section.
Fracture surface is flat, with no undulations.

Appendix 4: Effect of peeling angle on work to peel in hemp

block	plant	angle	peel work	block	plant	angle	peel work
1	1	0	248.61	2	5	0	216.66
1	1	15	212.90	2	5	15	210.49
1	1	30	191.34	2	5	30	190.40
1	1	45	191.49	2	5	45	220.31
1	1	60	217.83	2	5	60	226.49
1	1	75	261.51	2	5	75	237.80
1	1	85	248.99	2	5	85	236.38
1	2	0	211.50	2	6	0	266.76
1	2	15	184.00	2	6	15	278.92
1	2	30	190.44	2	6	30	273.09
1	2	45	209.96	2	6	45	267.19
1	2	60	255.49	2	6	60	233.22
1	2	75	293.22	2	6	75	225.37
1	2	85	350.23	2	6	85	209.23
1	3	0	210.73	2	7	0	168.13
1	3	15	187.00	2	7	15	174.84
1	3	30	181.11	2	7	30	190.50
1	3	45	189.36	2	7	45	213.30
1	3	60	192.61	2	7	60	230.53
1	3	75	220.61	2	7	75	297.07
1	3	85	261.22	2	7	85	351.64
1	5	0	144.01	3	2	0	277.96
1	5	15	137.78	3	2	15	203.67
1	5	30	131.05	3	2	30	206.31
1	5	45	139.28	3	2	45	208.58
1	5	60	150.91	3	2	60	229.75
1	5	75	179.78	3	2	75	247.27
1	5	85	194.21	3	2	85	231.10
1	6	0	168.81	3	4	0	213.92
1	6	15	159.72	3	4	15	217.54
1	6	30	134.04	3	4	30	177.15
1	6	45	148.37	3	4	45	186.30
1	6	60	161.84	3	4	60	216.36
1	6	75	201.16	3	4	75	216.94
1	6	85	181.12	3	4	85	238.75
2	1	0	151.33	3	5	0	133.92
2	1	15	156.47	3	5	15	146.14
2	1	30	154.31	3	5	30	143.26
2	1	45	169.67	3	5	45	148.21
2	1	60	167.90	3	5	60	136.96
2	1	75	182.61	3	5	75	153.28
2	1	85	240.95	3	5	85	169.53
2	2	0	226.84	3	6	0	189.19
2	2	15	233.56	3	6	15	169.07
2	2	30	212.35	3	6	30	168.54
2	2	45	195.17	3	6	45	159.93
2	2	60	219.68	3	6	60	184.74
2	2	75	254.34	3	6	75	232.38
2	2	85	259.43	3	6	85	250.42
2	4	0	67.84	3	7	0	114.06
2	4	15	82.30	3	7	15	115.76
2	4	30	106.69	3	7	30	116.14
2	4	45	107.29	3	7	45	137.22
2	4	60	163.52	3	7	60	136.18
2	4	75	193.51	3	7	75	163.21
2	4	85	217.01	3	7	85	154.83

Appendix 5: Effect of stem moisture content on work to peel in hemp

Block	Plant	Hrs in oven	M.C.	Peel Work	Block	Plant	Hrs in oven	M.C.	Peel Work
1	1	0	82.8	152.79	2	1	0	76.3	155.70
1	1	1	68.3	307.40	2	1	1	64.3	209.10
1	1	2	59.2	346.59	2	1	2	52.9	291.64
1	1	3	50.7	257.53	2	1	3	43.8	209.17
1	1	5	28.3	368.57	2	1	5	19.8	263.41
1	1	17	8.3	528.22	2	1	17	2.3	735.82
1	1	42	0.0	65.83	2	1	42	0.0	361.09
1	2	0	80.6	214.73	2	2	0	79.3	199.31
1	2	1	75.6	202.65	2	2	1	68.2	282.49
1	2	2	66.9	222.52	2	2	2	59.3	261.41
1	2	3	59.6	260.24	2	2	3	52.8	250.29
1	2	5	40.6	341.42	2	2	5	35.0	259.81
1	2	17	16.9	829.59	2	2	17	13.0	538.57
1	2	42	0.0	85.02	2	2	42	0.0	76.33
1	3	0	78.3	279.17	2	3	0	81.5	217.62
1	3	1	67.3	326.25	2	3	1	71.5	257.09
1	3	2	52.6	290.65	2	3	2	62.7	331.50
1	3	3	46.7	255.42	2	3	3	56.3	267.76
1	3	5	27.8	236.39	2	3	5	40.2	282.74
1	3	17	6.1	435.05	2	3	17	21.9	376.62
1	3	42	0.0	150.24	2	3	42	0.0	116.58
1	4	0	72.8	227.62	2	4	0	82.3	227.53
1	4	1	61.6	230.80	2	4	1	76.8	226.41
1	4	2	52.7	290.62	2	4	2	71.0	147.60
1	4	3	40.3	224.31	2	4	3	65.3	217.06
1	4	5	17.9	331.10	2	4	5	44.1	224.15
1	4	17	2.8	644.55	2	4	17	18.9	410.21
1	4	42	0.0	392.38	2	4	42	0.0	86.30
1	5	0	75.7	206.57	2	5	0	81.3	243.28
1	5	1	64.9	152.11	2	5	1	69.4	202.80
1	5	2	47.0	216.28	2	5	2	60.4	142.98
1	5	3	38.4	179.31	2	5	3	54.4	203.18
1	5	5	10.8	249.57	2	5	5	31.9	258.84
1	5	17	2.2	202.44	2	5	17	8.8	435.21
1	5	42	0.0	130.01	2	5	42	0.0	253.12
1	6	0	88.3	234.44	2	6	0	80.7	242.81
1	6	1	80.9	222.18	2	6	1	72.6	275.60
1	6	2	76.4	187.87	2	6	2	56.7	186.19
1	6	3	67.2	235.30	2	6	3	49.8	436.12
1	6	5	48.4	183.87	2	6	5	32.9	328.01
1	6	17	24.1	247.54	2	6	17	13.9	476.23
1	6	42	0.0	169.22	2	6	42	0.0	149.70
1	7	0	85.9	204.76	2	7	0	82.6	226.14
1	7	1	76.3	135.05	2	7	1	72.5	208.09
1	7	2	67.6	171.76	2	7	2	63.9	343.96
1	7	3	57.2	220.84	2	7	3	54.1	190.07
1	7	5	41.8	180.38	2	7	5	32.8	292.93
1	7	17	19.7	483.87	2	7	17	13.0	304.26
1	7	42	0.0	58.56	2	7	42	0.0	254.02

Appendix 5, continued: Effect of stem moisture content on work to peel in hemp

Block	Plant	Hrs in oven	M.C.	Peel Work
3	1	0	78.3	284.08
3	1	1	64.3	223.01
3	1	2	51.3	285.02
3	1	3	40.6	237.07
3	1	5	23.0	230.48
3	1	17	3.3	419.69
3	1	42	0.0	134.18
3	2	0	83.6	242.78
3	2	1	79.0	350.26
3	2	2	74.5	265.77
3	2	3	67.5	349.81
3	2	5	52.2	298.62
3	2	17	31.6	242.40
3	2	42	0.0	120.90
3	3	0	85.0	238.62
3	3	1	77.0	202.59
3	3	2	68.8	198.64
3	3	3	60.3	246.44
3	3	5	40.8	202.66
3	3	17	20.2	231.48
3	3	42	0.0	85.99
3	4	0	78.6	192.84
3	4	1	70.5	205.34
3	4	2	60.7	190.96
3	4	3	51.9	200.09
3	4	5	32.4	159.99
3	4	17	9.4	450.45
3	4	42	0.0	166.75
3	5	0	84.6	216.89
3	5	1	77.1	209.84
3	5	2	63.5	201.50
3	5	3	51.0	187.18
3	5	5	22.9	117.34
3	5	17	1.7	352.27
3	5	42	0.0	302.80
3	6	0	71.3	291.89
3	6	1	62.3	301.42
3	6	2	47.7	299.26
3	6	3	35.1	258.56
3	6	5	10.8	272.74
3	6	17	2.6	584.02
3	6	42	0.0	227.66
3	7	0	82.5	249.31
3	7	1	70.7	191.37
3	7	2	62.3	193.46
3	7	3	50.5	301.95
3	7	5	28.1	137.10
3	7	17	5.3	338.83
3	7	42	0.0	59.15

Appendix 6: Effect of stand retting on work to peel and work to tear in flax

Untreated control (weeks 0 – 3)

Week	Block	plant	% M.C.	Peel	Tear	Week	Block	plant	% M.C.	Peel	Tear
0	1	1	62.0	0.1807	0.0170	2	1	1	54.7	0.1996	0.0238
0	1	2	64.0	0.1625	0.0273	2	1	2	49.1	0.2054	0.0263
0	1	3	64.9	0.1337	0.0277	2	1	3	49.6	0.1991	0.0282
0	1	4	64.5	0.1372	0.0221	2	1	4	50.4	0.1943	0.0248
0	1	5	65.7	0.1349	0.0389	2	1	5	40.3	0.4665	0.0531
0	1	6	62.6	0.1458	0.0213	2	1	6	50.7	0.2115	0.0294
0	1	7	65.3	0.1933	0.0137	2	1	7	53.9	0.2217	0.0366
0	1	8	62.6	0.1597	0.0247	2	1	8	49.6	0.2494	0.0215
0	1	9	64.0	0.1362	0.0405	2	1	9	55.2	0.2516	0.0243
0	1	10	64.2	0.1659	0.0215	2	1	10	42.9	0.2341	0.0331
0	2	11	59.9	0.1342	0.0256	2	2	11	36.8	0.2103	0.0444
0	2	12	61.8	0.1607	0.0256	2	2	12	48.3	0.1303	0.0340
0	2	13	66.1	0.1408	0.0191	2	2	13	52.6	0.1697	0.0226
0	2	14	61.7	0.1551	0.0207	2	2	14	53.0	0.1932	0.0169
0	2	15	62.0	0.1917	0.0291	2	2	15	52.5	0.2511	0.0264
0	2	16	61.5	0.1521	0.0290	2	2	16	53.8	0.1624	0.0422
0	2	17	61.3	0.1745	0.0214	2	2	17	54.7	0.1901	0.0286
0	2	18	58.9	0.1734	0.0408	2	2	18	51.3	0.1650	0.0362
0	2	19	60.7	0.1771	0.0194	2	2	19	50.4	0.1357	0.0228
0	2	20	62.1	0.1500	0.0297	2	2	20	54.6	0.1676	0.0326
0	3	21	59.0	0.1508	0.0212	2	3	21	57.0	0.1506	0.0269
0	3	22	63.6	0.1887	0.0259	2	3	22	52.6	0.2170	0.0271
0	3	23	62.9	0.1715	0.0213	2	3	23	60.6	0.3038	0.0304
0	3	24	61.5	0.1797	0.0878	2	3	24	56.1	0.1380	0.0225
0	3	25	63.0	0.1443	0.0319	2	3	25	57.3	0.1501	0.0162
0	3	26	62.4	0.1836	0.0151	2	3	26	54.7	0.2184	0.0325
0	3	27	60.5	0.1470	0.0281	2	3	27	58.8	0.1381	0.0147
0	3	28	61.9	0.2259	0.0246	2	3	28	46.1	0.3472	0.0383
0	3	29	61.9	0.1358	0.0177	2	3	29	59.6	0.1570	0.0200
0	3	30	66.1	0.1496	0.0172	2	3	30	61.9	0.1180	0.0260
1	1	1	57.1	0.1817	0.0212	3	1	1	32.7	0.3048	0.0557
1	1	2	54.0	0.1594	0.0201	3	1	2	33.8	0.2272	0.0376
1	1	3	57.7	0.1797	0.0280	3	1	3	35.7	0.2743	0.0316
1	1	4	59.2	0.1837	0.0252	3	1	4	24.0	0.5092	0.0610
1	1	5	56.0	0.1370	0.0143	3	1	5	35.1	0.2672	0.0496
1	1	6	54.7	0.1889	0.0134	3	1	6	9.6	0.6366	0.0267
1	1	7	55.3	0.1972	0.0347	3	1	7	35.4	0.2493	0.0303
1	1	8	60.7	0.1838	0.0308	3	1	8	29.4	0.3278	0.0211
1	1	9	60.3	0.2021	0.0249	3	1	9	30.4	0.3657	0.0146
1	1	10	60.1	0.1831	0.0299	3	1	10	26.3	0.4650	0.0589
1	2	11	53.9	0.1563	0.0211	3	2	11	22.0	0.5020	0.0086
1	2	12	58.5	0.2303	0.0330	3	2	12	10.5	0.3716	0.0537
1	2	13	57.1	0.1600	0.0244	3	2	13	7.7	0.3591	0.0320
1	2	14	55.8	0.2113	0.0352	3	2	14	23.2	0.6191	0.0445
1	2	15	59.3	0.1582	0.0245	3	2	15	23.9	0.4613	0.0126
1	2	16	52.0	0.1614	0.0163	3	2	16	9.9	0.4694	0.0056
1	2	17	53.1	0.1750	0.0167	3	2	17	32.9	0.5316	0.0698
1	2	18	53.8	0.1463	0.0336	3	2	18	18.7	0.4314	0.0843
1	2	19	54.4	0.1406	0.0244	3	2	19	9.9	0.5503	0.0773
1	2	20	56.4	0.1963	0.0229	3	2	20	9.6	0.5383	0.0037
1	3	21	60.6	0.1405	0.0252	3	3	21	18.5	0.4466	0.0132
1	3	22	60.2	0.1743	0.0248	3	3	22	37.1	0.5089	0.0161
1	3	23	62.9	0.1464	0.0275	3	3	23	12.0	0.6553	0.0067
1	3	24	61.8	0.1180	0.0318	3	3	24	11.6	0.5370	0.0034
1	3	25	55.2	0.1491	0.0174	3	3	25	9.0	0.6013	0.0367
1	3	26	60.6	0.1795	0.0092	3	3	26	9.4	0.7046	0.0500
1	3	27	60.1	0.1687	0.0358	3	3	27	9.5	0.7014	0.0136
1	3	28	59.1	0.1363	0.0178	3	3	28	11.3	0.4737	0.0179
1	3	29	56.2	0.1747	0.0217	3	3	29	8.7	0.6828	0.0122
1	3	30	59.8	0.1935	0.0264	3	3	30	9.1	0.4874	0.0121

Appendix 6, continued: Untreated control (weeks 4 – 7)

Week	Block	plant	% M.C.	Peel	Tear	Week	Block	plant	% M.C.	Peel	Tear
4	1	1	12.5	0.5816	0.0696	6	1	1	11.6	0.0941	0.0086
4	1	2	15.0	0.3633	0.0727	6	1	2	12.3	0.7046	0.0409
4	1	3	10.3	0.3321	0.0119	6	1	3	11.7	0.2528	0.0054
4	1	4	10.5	0.3690	0.0133	6	1	4	11.4	0.1547	0.0088
4	1	5	10.6	0.3227	0.0225	6	1	5	11.6	0.6062	0.0136
4	1	6	11.4	0.4842	0.0133	6	1	6	12.3	0.0178	0.0018
4	1	7	11.0	0.4008	0.0114	6	1	7	11.6	0.4675	0.0036
4	1	8	27.3	0.3093	0.0272	6	1	8	12.3	0.5307	0.0290
4	1	9	10.4	0.5690	0.0261	6	1	9	12.2	0.2295	0.0097
4	1	10	13.4	0.5657	0.0163	6	1	10	11.7	0.4636	0.0246
4	2	11	26.6	0.3626	0.0266	6	2	11	10.6	0.0181	0.0030
4	2	12	11.7	0.3892	0.0338	6	2	12	12.2	0.3049	0.0340
4	2	13	17.0	0.4911	0.0446	6	2	13	11.4	0.2128	0.0094
4	2	14	14.7	0.4283	0.0107	6	2	14	12.7	0.3381	0.0074
4	2	15	12.0	0.4745	0.0086	6	2	15	12.1	0.2496	0.0117
4	2	16	10.0	0.5727	0.0355	6	2	16	12.1	0.4566	0.0162
4	2	17	12.2	0.4340	0.0355	6	2	17	12.0	0.1575	0.0184
4	2	18	12.1	0.2179	0.0092	6	2	18	11.9	0.3181	0.0315
4	2	19	11.6	0.5209	0.0245	6	2	19	13.5	0.1659	0.0152
4	2	20	11.0	0.7969	0.0080	6	2	20	12.9	0.2499	0.0187
4	3	21	18.9	0.5378	0.0220	6	3	21	11.9	0.5208	0.0062
4	3	22	16.9	0.2063	0.0288	6	3	22	11.4	0.1912	0.0142
4	3	23	16.4	0.4653	0.0235	6	3	23	12.6	0.2227	0.0101
4	3	24	16.7	0.4371	0.0068	6	3	24	12.2	0.7096	0.0641
4	3	25	13.7	0.3010	0.0207	6	3	25	12.1	0.3424	0.0138
4	3	26	12.6	0.3339	0.0148	6	3	26	12.1	0.4475	0.0057
4	3	27	14.8	0.4865	0.0119	6	3	27	12.3	0.3079	0.0097
4	3	28	12.0	0.3848	0.0538	6	3	28	10.6	0.0398	0.0052
4	3	29	12.9	0.3606	0.0435	6	3	29	12.2	0.1419	0.0253
4	3	30	14.7	0.6139	0.0173	6	3	30	12.0	0.3078	0.0173
5	1	1	10.0	0.4316	0.0028	7	1	1	12.4	0.0847	0.0107
5	1	2	11.2	0.5994	0.0363	7	1	2	12.1	0.1755	0.0341
5	1	3	10.5	0.2094	0.0207	7	1	3	13.3	0.1457	0.0131
5	1	4	11.8	0.4838	0.0294	7	1	4	12.6	0.3981	0.0661
5	1	5	12.0	0.2005	0.0096	7	1	5	12.6	0.1182	0.0537
5	1	6	11.1	0.7826	0.0660	7	1	6	12.6	0.2247	0.0199
5	1	7	10.8	0.4772	0.0200	7	1	7	12.5	0.2970	0.0256
5	1	8	11.0	0.3586	0.0124	7	1	8	11.3	0.2785	0.0048
5	1	9	13.1	0.4918	0.0618	7	1	9	13.2	0.1950	0.0109
5	1	10	11.9	0.4151	0.0063	7	1	10	12.7	0.4354	0.0051
5	2	11	13.1	0.5547	0.0221	7	2	11	15.1	0.0850	0.0143
5	2	12	11.7	0.4105	0.0068	7	2	12	14.0	0.2916	0.0018
5	2	13	10.9	0.2513	0.0123	7	2	13	12.5	0.3394	0.0569
5	2	14	9.0	0.4077	0.0074	7	2	14	12.6	0.1329	0.0068
5	2	15	8.8	0.6640	0.0142	7	2	15	13.2	0.1491	0.0114
5	2	16	11.4	0.3324	0.0126	7	2	16	12.3	0.1138	0.0055
5	2	17	7.8	0.5423	0.0072	7	2	17	15.7	0.1026	0.0069
5	2	18	9.1	0.5247	0.0467	7	2	18	12.5	0.2121	0.0303
5	2	19	10.8	0.3402	0.0150	7	2	19	12.2	0.0008	0.0019
5	2	20	11.5	0.3308	0.0069	7	2	20	12.0	0.1060	0.1310
5	3	21	10.5	0.3120	0.0088	7	3	21	12.4	0.1086	0.0145
5	3	22	12.4	0.6150	0.0387	7	3	22	12.3	0.0824	0.0071
5	3	23	11.2	0.5396	0.0291	7	3	23	11.9	0.0442	0.0192
5	3	24	11.4	0.6964	0.0126	7	3	24	12.5	0.0204	0.0027
5	3	25	11.0	0.4041	0.0059	7	3	25	18.9	0.4266	0.0579
5	3	26	11.5	0.7638	0.0107	7	3	26	14.1	0.0115	0.0088
5	3	27	11.0	0.0616	0.0077	7	3	27	12.1	0.0093	0.0213
5	3	28	10.1	0.0726	0.0063	7	3	28	11.8	0.1441	0.0124
5	3	29	9.9	0.6655	0.0071	7	3	29	12.6	0.2316	0.0525
5	3	30	10.2	0.1821	0.0106	7	3	30	12.8	0.1692	0.0073

Appendix 6, continued: Untreated control (week 8)

Week	Block	plant	% M.C.	Peel	Tear
8	1	1	9.4	0.1012	0.0217
8	1	2	9.1	0.0004	0.0037
8	1	3	14.9	0.0682	0.0068
8	1	4	10.5	0.1367	0.0084
8	1	5	10.6	0.2825	0.0042
8	1	6	9.9	0.2929	0.0085
8	1	7	9.0	0.0873	0.0446
8	1	8	9.4	0.1048	0.0098
8	1	9	10.1	0.2976	0.0145
8	1	10	9.6	0.1158	0.0100
8	2	11	8.9	0.0127	0.0048
8	2	12	9.1	0.0003	0.0497
8	2	13	9.2	0.1152	0.0069
8	2	14	10.1	0.2219	0.0284
8	2	15	8.8	0.5631	0.0068
8	2	16	7.9	0.4231	0.0134
8	2	17	9.0	0.0279	0.0005
8	2	18	9.3	0.2185	0.0527
8	2	19	9.6	0.2236	0.0070
8	2	20	9.2	0.1951	0.0325
8	3	21	9.5	0.2505	0.0348
8	3	22	9.0	0.1095	0.0229
8	3	23	9.5	0.3608	0.0162
8	3	24	8.8	0.2071	0.0288
8	3	25	9.0	0.0001	0.0016
8	3	26	9.6	0.1815	0.0075
8	3	27	9.3	0.0489	0.0301
8	3	28	7.9	0.1744	0.0117
8	3	29	8.6	0.2673	0.0083
8	3	30	8.8	0.1536	0.0141

Appendix 6 continued: Touchdown (weeks 0 - 3)

Week	Block	plant	% M.C.	Peel	Tear	Week	Block	plant	% M.C.	Peel	Tear
0	1	1	62.0	0.1807	0.0170	2	1	1	58.6	0.1536	0.0163
0	1	2	64.0	0.1625	0.0273	2	1	2	58.3	0.1771	0.0273
0	1	3	64.9	0.1337	0.0277	2	1	3	58.1	0.1762	0.0334
0	1	4	64.5	0.1372	0.0221	2	1	4	56.7	0.1390	0.0215
0	1	5	65.7	0.1349	0.0389	2	1	5	59.8	0.1455	0.0185
0	1	6	62.6	0.1458	0.0213	2	1	6	59.2	0.1843	0.0316
0	1	7	65.3	0.1933	0.0137	2	1	7	55.7	0.1451	0.0170
0	1	8	62.6	0.1597	0.0247	2	1	8	55.8	0.1667	0.0339
0	1	9	64.0	0.1362	0.0405	2	1	9	53.0	0.1327	0.0311
0	1	10	64.2	0.1659	0.0215	2	1	10	61.0	0.1468	0.0339
0	2	11	59.9	0.1342	0.0256	2	2	11	59.3	0.1871	0.0246
0	2	12	61.8	0.1607	0.0256	2	2	12	58.0	0.1663	0.0251
0	2	13	66.1	0.1408	0.0191	2	2	13	61.6	0.1204	0.0172
0	2	14	61.7	0.1551	0.0207	2	2	14	60.3	0.1868	0.0228
0	2	15	62.0	0.1917	0.0291	2	2	15	58.2	0.1583	0.0290
0	2	16	61.5	0.1521	0.0290	2	2	16	61.0	0.1496	0.0254
0	2	17	61.3	0.1745	0.0214	2	2	17	59.6	0.0943	0.0295
0	2	18	58.9	0.1734	0.0408	2	2	18	61.3	0.1989	0.0167
0	2	19	60.7	0.1771	0.0194	2	2	19	57.4	0.1642	0.0285
0	2	20	62.1	0.1500	0.0297	2	2	20	61.4	0.1604	0.0157
0	3	21	59.0	0.1508	0.0212	2	3	21	63.7	0.1428	0.0233
0	3	22	63.6	0.1887	0.0259	2	3	22	52.8	0.1824	0.0160
0	3	23	62.9	0.1715	0.0213	2	3	23	55.4	0.1594	0.0259
0	3	24	61.5	0.1797	0.0878	2	3	24	60.1	0.1574	0.0263
0	3	25	63.0	0.1443	0.0319	2	3	25	57.5	0.1797	0.0297
0	3	26	62.4	0.1836	0.0151	2	3	26	58.6	0.1511	0.0213
0	3	27	60.5	0.1470	0.0281	2	3	27	34.7	0.2392	0.0131
0	3	28	61.9	0.2259	0.0246	2	3	28	58.6	0.1988	0.0392
0	3	29	61.9	0.1358	0.0177	2	3	29	58.9	0.1813	0.0281
0	3	30	66.1	0.1496	0.0172	2	3	30	54.8	0.1817	0.0313
1	1	1	56.2	0.1697	0.0212	3	1	1	8.5	0.6704	0.0058
1	1	2	58.2	0.1876	0.0268	3	1	2	12.0	0.5648	0.0092
1	1	3	59.4	0.1633	0.0365	3	1	3	8.5	0.5512	0.0042
1	1	4	58.4	0.1837	0.0247	3	1	4	13.4	0.5846	0.0660
1	1	5	54.7	0.1614	0.0208	3	1	5	8.1	0.7116	0.0066
1	1	6	59.4	0.1467	0.0259	3	1	6	7.8	0.5243	0.0041
1	1	7	54.3	0.1466	0.0200	3	1	7	9.9	0.5422	0.0334
1	1	8	56.8	0.1709	0.0243	3	1	8	10.9	0.5621	0.0347
1	1	9	61.2	0.1472	0.0325	3	1	9	12.1	0.4135	0.0701
1	1	10	55.5	0.2085	0.0385	3	1	10	12.3	0.4920	0.0135
1	2	11	61.1	0.1543	0.0221	3	2	11	13.6	0.4589	0.0051
1	2	12	59.2	0.1733	0.0209	3	2	12	8.6	0.3752	0.0243
1	2	13	54.0	0.1792	0.0324	3	2	13	10.6	0.5070	0.0038
1	2	14	56.5	0.1635	0.0266	3	2	14	24.2	0.7034	0.0540
1	2	15	57.3	0.1491	0.0178	3	2	15	12.7	0.3816	0.0440
1	2	16	64.0	0.1283	0.0154	3	2	16	28.3	0.6528	0.0145
1	2	17	56.3	0.1501	0.0208	3	2	17	24.0	0.5082	0.0289
1	2	18	55.4	0.1799	0.0250	3	2	18	24.4	0.3928	0.0073
1	2	19	52.9	0.1821	0.0195	3	2	19	13.3	0.6189	0.0476
1	2	20	57.1	0.1617	0.0273	3	2	20	31.6	0.5894	0.0704
1	3	21	59.3	0.1557	0.0265	3	3	21	11.1	0.6777	0.0095
1	3	22	57.8	0.1466	0.0187	3	3	22	10.2	0.7506	0.0331
1	3	23	61.2	0.1311	0.0167	3	3	23	9.5	0.5673	0.0097
1	3	24	57.9	0.1380	0.0149	3	3	24	7.0	0.4917	0.0078
1	3	25	56.9	0.1551	0.0272	3	3	25	20.3	0.4844	0.0084
1	3	26	58.1	0.3857	0.0401	3	3	26	9.5	0.5508	0.0081
1	3	27	59.0	0.1662	0.0277	3	3	27	10.1	0.5539	0.0309
1	3	28	60.6	0.1448	0.0261	3	3	28	11.5	0.5844	0.1329
1	3	29	52.7	0.1540	0.0149	3	3	29	8.2	0.5481	0.0151
1	3	30	55.3	0.1219	0.0164	3	3	30	8.1	0.4500	0.0124

Appendix 6, continued: Touchdown (weeks 4 – 7)

Week	Block	plant	% M.C.	Peel	Tear	Week	Block	plant	% M.C.	Peel	Tear
4	1	1	11.2	0.2654	0.0075	6	1	1	5.9	0.0874	0.0074
4	1	2	10.8	0.2703	0.0587	6	1	2	7.1	0.1408	0.0179
4	1	3	11.4	0.5306	0.0228	6	1	3	8.8	0.2384	0.0146
4	1	4	13.0	0.4270	0.0153	6	1	4	8.2	0.2253	0.0075
4	1	5	10.7	0.2475	0.0246	6	1	5	8.2	0.2603	0.0307
4	1	6	10.7	0.3800	0.0241	6	1	6	9.4	0.2444	0.0129
4	1	7	12.2	0.3158	0.0317	6	1	7	9.5	0.1748	0.0102
4	1	8	11.6	0.2936	0.0102	6	1	8	9.4	0.3232	0.0119
4	1	9	12.8	0.5266	0.0870	6	1	9	9.3	0.1947	0.0095
4	1	10	11.3	0.3201	0.0126	6	1	10	7.4	0.2570	0.0060
4	2	11	12.2	0.2009	0.0220	6	2	11	9.0	0.6784	0.0369
4	2	12	14.6	0.3556	0.0061	6	2	12	10.9	0.1900	0.0206
4	2	13	17.9	0.4422	0.0049	6	2	13	9.6	0.3358	0.0880
4	2	14	13.7	0.6811	0.0385	6	2	14	9.0	0.3795	0.0257
4	2	15	35.7	0.5821	0.0482	6	2	15	10.7	0.0959	0.0070
4	2	16	18.9	0.2599	0.0260	6	2	16	9.6	0.5294	0.0090
4	2	17	13.5	0.3976	0.0126	6	2	17	9.6	0.4983	0.0113
4	2	18	12.6	0.4062	0.0081	6	2	18	9.3	0.3863	0.0094
4	2	19	16.4	0.5471	0.0332	6	2	19	8.9	0.2034	0.0220
4	2	20	13.3	0.4300	0.0255	6	2	20	10.2	0.2503	0.0076
4	3	21	12.8	0.3657	0.0142	6	3	21	11.0	0.2704	0.0156
4	3	22	23.5	0.1697	0.0518	6	3	22	12.0	0.2533	0.0361
4	3	23	11.7	0.1717	0.0105	6	3	23	10.4	0.2013	0.0123
4	3	24	12.7	0.1271	0.0107	6	3	24	11.0	0.2274	0.0079
4	3	25	13.1	0.3858	0.0537	6	3	25	9.1	0.3446	0.0073
4	3	26	11.0	0.2586	0.0089	6	3	26	9.8	0.1937	0.0137
4	3	27	11.4	0.3473	0.0073	6	3	27	10.3	0.1421	0.0060
4	3	28	11.4	0.4153	0.0434	6	3	28	10.4	0.2453	0.0625
4	3	29	10.3	0.2321	0.0276	6	3	29	11.2	0.4158	0.0288
4	3	30	11.2	0.3058	0.0324	6	3	30	10.0	0.2321	0.0085
5	1	1	9.6	0.1453	0.0590	7	1	1	11.9	0.2841	0.0141
5	1	2	10.8	0.1953	0.0155	7	1	2	13.8	0.1237	0.0097
5	1	3	10.4	0.2807	0.0336	7	1	3	11.3	0.1331	0.0062
5	1	4	12.9	0.1428	0.0366	7	1	4	12.1	0.2227	0.0069
5	1	5	10.3	0.3277	0.0334	7	1	5	12.3	0.1757	0.0106
5	1	6	9.1	0.1878	0.0057	7	1	6	12.2	0.1630	0.0097
5	1	7	11.3	0.2801	0.0064	7	1	7	12.2	0.0995	0.0063
5	1	8	11.4	0.1346	0.0095	7	1	8	12.5	0.2665	0.0141
5	1	9	10.9	0.3899	0.0093	7	1	9	12.5	0.1997	0.0081
5	1	10	10.4	0.1723	0.0168	7	1	10	12.2	0.2547	0.0110
5	2	11	10.3	0.4833	0.0236	7	2	11	12.2	0.1257	0.0061
5	2	12	19.2	0.2237	0.0336	7	2	12	13.5	0.1946	0.0255
5	2	13	12.2	0.2258	0.0089	7	2	13	12.2	0.1591	0.0101
5	2	14	10.3	0.3483	0.0389	7	2	14	11.4	0.0197	0.0124
5	2	15	10.7	0.3994	0.0310	7	2	15	12.3	0.3149	0.0274
5	2	16	10.7	0.1941	0.0087	7	2	16	12.5	0.3707	0.0387
5	2	17	11.4	0.2044	0.0175	7	2	17	12.3	0.1525	0.0148
5	2	18	10.6	0.3767	0.0146	7	2	18	12.6	0.1652	0.0281
5	2	19	11.3	0.3935	0.0046	7	2	19	12.7	0.2721	0.0173
5	2	20	10.5	0.2291	0.0110	7	2	20	14.0	0.0051	0.0021
5	3	21	11.5	0.2923	0.0137	7	3	21	9.7	0.0927	0.0076
5	3	22	10.2	0.1083	0.0049	7	3	22	9.8	0.0177	0.0072
5	3	23	11.1	0.1608	0.0083	7	3	23	9.5	0.2860	0.0130
5	3	24	10.1	0.1295	0.0070	7	3	24	9.2	0.1196	0.0075
5	3	25	10.8	0.3533	0.0161	7	3	25	10.6	0.1295	0.0065
5	3	26	10.5	0.0624	0.0528	7	3	26	11.0	0.2334	0.0081
5	3	27	11.4	0.2107	0.0060	7	3	27	10.5	0.1740	0.0133
5	3	28	10.9	0.3457	0.0147	7	3	28	10.8	0.3939	0.0031
5	3	29	11.6	0.1564	0.0036	7	3	29	10.1	0.1268	0.0102
5	3	30	9.6	0.2905	0.0114	7	3	30	9.5	0.1420	0.0202

Appendix 6, continued: Touchdown (week 8)

Week	Block	plant	% M.C.	Peel	Tear
8	1	1	8.1	0.0848	0.0221
8	1	2	9.6	0.0966	0.0183
8	1	3	9.3	0.0434	0.0053
8	1	4	10.0	0.4188	0.0103
8	1	5	9.9	0.0855	0.0056
8	1	6	10.0	0.0749	0.0054
8	1	7	11.3	0.1479	0.0064
8	1	8	10.7	0.1706	0.0061
8	1	9	10.8	0.0194	0.0200
8	1	10	10.7	0.1126	0.0070
8	2	11	7.1	0.0002	0.0019
8	2	12	12.0	0.0598	0.0122
8	2	13	9.3	0.7222	0.0234
8	2	14	9.1	0.1275	0.0127
8	2	15	9.4	0.1342	0.0156
8	2	16	8.5	0.2227	0.0089
8	2	17	7.9	0.0004	0.0048
8	2	18	8.6	0.1265	0.0087
8	2	19	8.3	0.0072	0.0025
8	2	20	9.3	0.1919	0.0126
8	3	21	7.0	0.0004	0.0032
8	3	22	7.7	0.1621	0.0060
8	3	23	6.6	0.1121	0.0556
8	3	24	5.0	0.0004	0.0027
8	3	25	6.3	0.0005	0.0011
8	3	26	9.0	0.1322	0.0054
8	3	27	8.7	0.0499	0.0186
8	3	28	7.4	0.1168	0.0038
8	3	29	8.1	0.0722	0.0067
8	3	30	7.0	0.0100	0.0078

Appendix 7: Effect of dew-retting in pulled hemp

day	block	plant	Peel Work	MC %	day	block	plant	Peel Work	MC %
0	1	1	167.17	84.0	14	2	12	183.20	52.2
0	1	2	181.24	79.3	14	2	13	151.75	27.2
0	1	3	210.73	77.0	14	2	14	159.73	44.8
0	1	4	141.70	71.4	14	3	15	182.02	22.2
0	1	5	155.16	74.2	14	3	16	266.60	48.5
0	1	6	218.62	87.2	14	3	17	194.18	46.5
0	1	7	165.01	85.6	14	3	18	217.39	49.3
0	2	8	158.51	73.5	14	3	19	137.05	54.8
0	2	9	155.47	78.6	14	3	20	259.40	39.3
0	2	10	150.57	79.7	14	3	21	111.01	47.4
0	2	11	247.30	81.0	28	1	1	289.47	11.8
0	2	12	151.00	81.2	28	1	2	105.01	10.9
0	2	13	180.88	78.2	28	1	3	65.58	10.0
0	2	14	220.68	80.8	28	1	4	94.09	11.5
0	3	15	231.40	78.1	28	1	5	212.24	13.6
0	3	16	234.32	83.3	28	1	6	103.56	27.3
0	3	17	194.72	84.1	28	1	7	63.94	11.5
0	3	18	196.29	76.2	28	2	8	42.99	13.6
0	3	19	169.21	82.9	28	2	9	111.77	14.0
0	3	20	217.59	68.3	28	2	10	150.24	12.0
0	3	21	160.60	82.0	28	2	11	89.90	14.9
7	1	1	251.45	55.8	28	2	12	75.99	41.2
7	1	2	124.98	60.6	28	2	13	184.30	33.3
7	1	3	195.09	57.1	28	2	14	80.22	15.0
7	1	4	364.45	42.9	28	3	15	92.49	30.1
7	1	5	171.89	62.8	28	3	16	205.28	27.0
7	1	6	174.02	57.1	28	3	17	97.64	11.6
7	1	7	277.61	50.0	28	3	18	147.84	31.3
7	2	8	264.00	44.3	28	3	19	78.29	15.5
7	2	9	154.56	70.0	28	3	20	111.72	11.7
7	2	10	189.60	55.6	28	3	21	63.90	11.0
7	2	11	198.27	53.7	35	1	1	50.73	12.5
7	2	12	182.17	63.5	35	1	2	220.12	11.3
7	2	13	237.75	54.5	35	1	3	115.03	11.3
7	2	14	234.67	46.8	35	1	4	161.01	10.3
7	3	15	242.69	56.4	35	1	5	93.53	13.6
7	3	16	260.06	56.0	35	1	6	180.14	13.1
7	3	17	244.34	51.0	35	1	7	87.41	11.5
7	3	18	183.60	62.5	35	2	8	105.82	10.2
7	3	19	132.33	64.4	35	2	9	302.47	11.5
7	3	20	169.63	66.3	35	2	10	65.25	11.5
7	3	21	171.28	53.6	35	2	11	142.05	11.3
14	1	1	149.18	53.8	35	2	12	39.29	11.0
14	1	2	132.80	29.8	35	2	13	49.88	10.1
14	1	3	190.95	52.3	35	2	14	106.19	12.0
14	1	4	90.50	57.9	35	3	15	151.09	9.9
14	1	5	167.82	61.4	35	3	16	112.25	10.8
14	1	6	157.95	45.2	35	3	17	124.89	13.6
14	1	7	224.74	56.4	35	3	18	96.76	11.5
14	2	8	184.25	40.3	35	3	19	116.09	9.7
14	2	9	122.21	46.1	35	3	20	96.77	11.1
14	2	10	161.26	46.5	35	3	21	56.45	11.1
14	2	11	137.47	54.0					

Appendix 8: Proportion of shive removed from enzyme-retted flax

Hours	start	roll 1	roll 2	roll 3	roll 4	final
16	0	2.08	2.50	18.75	38.75	100
16	0	3.91	28.26	51.74	75.65	100
16	0	4.51	12.85	50.35	80.90	100
16	0	6.54	76.91	84.10	93.90	100
16	0	3.43	66.01	88.89	98.53	100
16	0	6.44	35.34	87.94	85.24	100
16	0	3.53	19.79	57.24	64.49	100
16	0	4.45	37.72	62.67	72.73	100
16	0	6.91	37.28	71.11	80.49	100
16	0	1.92	47.28	77.00	92.97	100
20	0	0.40	3.17	21.03	41.27	100
20	0	8.66	65.75	85.43	95.28	100
20	0	13.31	58.36	90.37	89.80	100
20	0	12.79	50.91	91.64	94.78	100
20	0	39.87	75.50	89.98	90.20	100
20	0	1.01	73.24	85.51	93.36	100
20	0	16.27	78.76	85.47	93.42	100
20	0	6.16	58.33	64.49	71.74	100
20	0	25.48	39.29	80.71	85.95	100
20	0	10.56	83.82	107.33	109.88	100
24	0	0.00	0.00	7.41	24.07	100
24	0	3.17	22.22	39.15	53.44	100
24	0	4.69	14.55	56.81	67.61	100
24	0	10.51	69.44	85.82	95.35	100
24	0	8.00	66.95	86.95	93.89	100
24	0	7.99	42.57	80.86	87.92	100
24	0	11.73	70.00	95.58	100.77	100
24	0	9.87	72.71	81.86	94.34	100
24	0	9.90	78.24	92.47	96.79	100
24	0	10.14	93.84	94.93	98.90	100
40	0	6.78	13.56	42.80	85.17	100
40	0	0.00	53.16	78.16	91.77	100
40	0	2.05	27.69	61.54	74.36	100
40	0	6.81	48.51	80.64	92.77	100
40	0	7.29	77.94	91.09	96.36	100
40	0	1.49	68.43	92.89	96.03	100
40	0	6.38	49.57	79.79	92.34	100
40	0	4.66	69.18	78.85	85.30	100
40	0	5.06	46.20	85.44	97.47	100
40	0	0.60	38.81	71.34	89.55	100
44	0	0.00	2.86	17.71	43.43	100
44	0	2.14	14.96	51.28	74.79	100
44	0	8.36	63.11	74.64	78.67	100
44	0	3.72	84.88	89.77	96.74	100
44	0	1.98	83.08	85.27	91.43	100
44	0	9.61	86.48	102.44	97.56	100
44	0	28.30	85.10	98.50	100.00	100
44	0	5.36	70.16	87.18	92.07	100
44	0	18.95	68.77	82.13	86.82	100
44	0	9.04	83.78	90.69	93.09	100

Appendix 9: Proportion of shive removed from stand retted flax

Untreated control (weeks 0 – 11)

date	block	plant	diam	start	roll 1	roll 2	roll 3	roll 4	final
0	1	1	1.59	0	0.00	44.19	79.46	88.37	100
0	1	2	2.26	0	1.69	47.37	76.32	86.84	100
0	1	3	1.91	0	4.89	89.24	95.11	98.29	100
0	1	4	1.96	0	1.47	42.65	88.24	92.89	100
0	1	5	1.89	0	0.23	53.81	85.22	93.76	100
0	2	6	2.32	0	1.08	44.75	77.31	94.91	100
0	2	7	2.03	0	0.48	46.19	86.98	94.29	100
0	2	8	1.60	0	4.04	46.46	67.34	75.76	100
0	2	9	1.73	0	5.50	28.52	70.45	79.04	100
0	2	10	1.51	0	4.76	63.95	87.76	92.86	100
0	3	11	2.31	0	0.87	44.89	84.92	90.99	100
0	3	12	2.13	0	1.96	46.76	86.25	92.53	100
0	3	13	2.18	0	6.85	58.47	74.19	82.06	100
0	3	14	1.38	0	4.72	25.32	63.95	79.83	100
0	3	15	1.96	0	18.87	70.89	90.57	95.15	100
2	1	1	1.75	0	0.65	8.82	11.44	15.36	100
2	1	2	2.61	0	0.40	135.22	33.11	53.69	100
2	1	3	2.55	0	1.01	8.45	37.20	56.49	100
2	1	4	1.87	0	1.03	8.44	15.84	25.72	100
2	1	5	1.76	0	0.00	0.34	1.01	5.07	100
2	2	6	2.02	0	0.70	1.17	6.07	14.95	100
2	2	7	2.27	0	4.72	13.95	24.95	36.15	100
2	2	8	2.67	0	0.43	1.57	7.42	45.22	100
2	2	9	1.54	0	0.81	2.43	5.26	8.50	100
2	2	10	2.11	0	2.42	5.27	14.07	21.98	100
2	3	11	1.42	0	2.04	13.47	22.86	28.16	100
2	3	12	1.41	0	2.63	9.65	15.35	25.88	100
2	3	13	1.74	0	2.06	20.62	48.97	65.98	100
2	3	14	1.54	0	3.99	11.59	19.93	28.62	100
2	3	15	1.74	0	7.49	16.91	29.23	45.65	100
4	1	1	1.73	0	0.98	5.25	18.69	27.54	100
4	1	2	2.16	0	1.05	7.13	14.47	23.90	100
4	1	3	2.20	0	4.31	32.58	44.57	60.49	100
4	1	4	1.95	0	2.39	9.13	21.96	33.48	100
4	1	5	2.18	0	2.05	11.95	32.59	46.25	100
4	2	6	1.58	0	0.00	9.09	17.27	24.55	100
4	2	7	1.68	0	0.00	30.58	63.41	68.42	100
4	2	8	1.54	0	5.82	10.96	17.81	20.89	100
4	2	9	2.09	0	0.00	20.52	49.20	68.70	100
4	2	10	1.84	0	3.73	36.57	50.75	65.42	100
4	3	11	1.98	0	3.78	21.66	62.22	72.54	100
4	3	12	2.43	0	0.74	15.91	33.40	69.86	100
4	3	13	2.08	0	2.17	22.78	59.86	68.54	100
4	3	14	2.11	0	11.68	51.95	77.84	85.18	100
4	3	15	2.00	0	1.56	20.82	43.19	63.62	100
6	1	1	2.16	0	0.00	10.18	19.96	27.54	100
6	1	2	2.42	0	0.28	16.37	50.34	71.94	100
6	1	3	2.15	0	0.00	1.19	8.83	16.30	100
6	1	4	2.82	0	0.00	0.00	1.52	3.64	100
6	1	5	2.54	0	0.00	5.05	21.88	44.59	100
6	2	6	2.14	0	0.00	4.31	33.61	60.00	100
6	2	7	2.11	0	0.00	10.31	29.37	43.18	100
6	2	8	2.23	0	1.21	14.78	29.86	50.23	100

6	2	9	1.90	0	0.94	12.43	40.30	70.06	100
6	2	10	1.83	0	0.00	3.74	13.63	18.24	100
6	3	11	1.20	0	2.41	7.23	19.68	24.90	100
6	3	12	1.83	0	0.17	0.69	57.61	18.51	100
6	3	13	1.83	0	0.00	7.89	26.71	38.24	100
6	3	14	1.50	0	1.03	10.82	15.46	21.91	100
6	3	15	1.45	0	1.04	7.77	12.18	16.58	100
8	1	1	1.84	0	1.34	56.30	68.10	79.62	100
8	1	2	2.10	0	1.64	42.54	79.35	87.32	100
8	1	3	2.26	0	3.38	30.18	44.59	71.62	100
8	1	4	2.17	0	3.82	43.15	68.99	77.98	100
8	1	5	2.10	0	2.22	28.83	64.52	86.69	100
8	2	6	1.87	0	2.96	22.62	51.16	66.17	100
8	2	7	2.00	0	0.94	20.90	52.17	72.88	100
8	2	8	1.68	0	2.91	18.02	32.27	58.43	100
8	2	9	1.90	0	4.14	26.93	79.85	88.89	100
8	2	10	1.63	0	0.00	15.15	19.01	48.21	100
8	3	11	1.44	0	1.27	21.10	48.52	50.63	100
8	3	12	1.87	0	0.41	31.57	60.90	78.00	100
8	3	13	1.83	0	2.48	19.14	42.12	55.63	100
8	3	14	1.70	0	0.00	30.73	57.68	71.71	100
8	3	15	1.63	0	0.62	15.48	34.06	41.80	100
11	1	1	1.90	0	2.69	26.65	43.60	69.42	100
11	1	2	2.24	0	0.40	10.28	48.59	62.50	100
11	1	3	2.17	0	0.19	9.11	55.79	64.33	100
11	1	4	2.36	0	0.30	65.67	86.06	94.00	100
11	1	5	1.85	0	0.00	11.76	55.59	61.18	100
11	2	6	1.57	0	2.56	40.06	67.63	77.88	100
11	2	7	1.67	0	2.64	62.38	81.85	87.13	100
11	2	8	1.60	0	2.72	48.34	66.16	76.44	100
11	2	9	1.44	0	2.80	29.91	66.36	85.98	100
11	2	10	1.72	0	2.28	32.76	55.27	68.38	100
11	3	11	1.87	0	0.27	27.96	60.48	65.32	100
11	3	12	2.40	0	0.14	88.44	96.68	98.70	100
11	3	13	2.13	0	3.50	45.79	78.38	85.21	100
11	3	14	2.24	0	0.49	41.30	77.56	84.72	100
11	3	15	1.91	0	4.43	48.36	74.18	77.46	100

Appendix 9 continued: Proportion of shive removed from desiccated flax samples Quattro (weeks 2 – 11)

date	block	plant	diam	start	roll 1	roll 2	roll 3	roll 4	final
2	1	1	1.62	0	0.28	3.06	3.33	9.44	100
2	1	2	1.66	0	0.32	0.32	0.97	2.58	100
2	1	3	2.29	0	0.44	0.88	26.40	34.07	100
2	1	4	1.96	0	0.38	5.92	16.98	41.41	100
2	1	5	1.52	0	0.59	1.17	2.64	5.57	100
2	2	6	1.94	0	0.25	13.68	23.63	34.58	100
2	2	7	1.60	0	2.48	3.41	6.50	10.53	100
2	2	8	1.95	0	2.24	19.07	34.58	46.73	100
2	2	9	1.67	0	0.52	1.55	5.68	6.46	100
2	2	10	1.14	0	0.00	1.91	1.91	1.91	100
2	3	11	1.75	0	0.00	0.00	4.90	8.07	100
2	3	12	2.10	0	0.31	0.46	10.37	15.94	100
2	3	13	1.99	0	0.53	5.52	23.67	36.83	100
2	3	14	0.86	0	0.84	2.10	2.10	4.62	100
2	3	15	1.85	0	0.00	4.32	5.58	16.19	100
4	1	1	2.60	0	1.04	8.84	43.87	78.07	100
4	1	2	2.07	0	5.52	45.33	81.52	96.57	100
4	1	3	1.87	0	7.69	62.98	83.89	91.59	100
4	1	4	2.29	0	1.34	55.97	84.54	89.75	100
4	1	5	2.05	0	1.89	16.67	77.08	85.61	100
4	2	6	1.38	0	2.56	4.10	6.15	11.28	100
4	2	7	1.89	0	0.00	0.32	2.88	9.27	100
4	2	8	1.84	0	0.53	2.91	3.44	4.76	100
4	2	9	1.98	0	1.42	17.97	38.53	57.92	100
4	2	10	1.66	0	0.97	3.14	9.18	16.43	100
4	3	11	1.32	0	1.27	1.27	3.82	6.37	100
4	3	12	1.26	0	0.00	0.99	2.46	7.39	100
4	3	13	1.72	0	2.70	9.70	17.52	24.26	100
4	3	14	1.74	0	0.48	14.98	34.30	52.90	100
4	3	15	2.48	0	0.38	7.75	58.58	82.08	100
6	1	1	1.44	0	0.44	7.42	9.61	25.33	100
6	1	2	2.24	0	0.72	4.88	35.58	72.60	100
6	1	3	2.06	0	0.53	46.81	68.97	88.48	100
6	1	4	1.59	0	0.81	3.23	6.85	8.06	100
6	1	5	1.95	0	0.65	94.64	78.57	88.15	100
6	2	6	1.71	0	0.72	10.02	18.14	25.78	100
6	2	7	1.77	0	0.38	2.29	11.47	26.96	100
6	2	8	1.69	0	3.38	9.66	32.37	52.90	100
6	2	9	1.50	0	0.94	11.95	22.64	28.93	100
6	2	10	1.43	0	2.43	9.42	23.10	32.83	100
6	3	11	2.10	0	0.36	0.00	10.07	29.32	100
6	3	12	1.58	0	0.00	0.38	7.28	9.96	100
6	3	13	2.10	0	0.34	20.14	40.44	51.10	100
6	3	14	1.73	0	0.91	1.52	7.60	10.64	100
6	3	15	1.90	0	1.92	3.19	21.73	34.19	100
8	1	1	1.61	0	66.67	90.14	100.29	99.71	100
8	1	2	2.00	0	15.60	80.64	95.86	95.68	100
8	1	3	1.76	0	100.00	100.00	100.00	100.00	100
8	1	4	1.53	0	99.12	99.41	99.41	99.71	100
8	1	5	1.58	0	3.64	81.82	85.00	90.45	100
8	2	6	1.94	0	31.24	91.01	96.18	96.85	100
8	2	7	1.95	0	69.39	82.80	93.00	98.83	100
8	2	8	1.63	0	88.41	94.57	97.83	99.28	100
8	2	9	1.82	0	79.69	91.08	96.62	98.15	100

8	2	10	1.41	0	50.90	83.78	86.49	88.74	100
8	3	11	1.83	0	6.52	55.65	85.22	92.39	100
8	3	12	1.87	0	1.49	35.61	60.98	73.56	100
8	3	13	2.15	0	0.91	14.31	34.96	50.54	100
8	3	14	1.56	0	8.70	40.87	72.61	86.52	100
8	3	15	2.08	0	2.80	20.75	58.28	89.74	100
11	1	1	2.05	0	98.35	99.59	99.79	99.79	100
11	1	2	1.80	0	98.74	100.00	100.00	100.00	100
11	1	3	2.04	0	59.48	94.81	98.80	100.00	100
11	1	4	1.80	0	78.97	93.36	100.00	100.00	100
11	1	5	2.25	0	98.72	99.82	100.00	100.00	100
11	2	6	1.82	0	1.51	28.29	67.60	83.80	100
11	2	7	1.26	0	0.00	35.16	79.69	83.59	100
11	2	8	1.60	0	99.22	100.00	100.00	100.00	100
11	2	9	1.68	0	99.35	100.00	100.00	100.00	100
11	2	10	1.93	0	26.42	80.00	98.52	98.52	100
11	3	11	2.05	0	2.40	43.89	59.39	74.45	100
11	3	12	2.03	0	3.00	14.59	62.66	84.55	100
11	3	13	1.62	0	90.11	92.02	94.68	94.68	100
11	3	14	1.54	0	95.11	98.22	100.00	100.00	100
11	3	15	2.66	0	0.73	11.53	33.14	40.88	100

Appendix 9, continued: Proportion of shive removed from pulled flax samples
Pulled and stand-retted (weeks 2 – 8)

date	block	plant	diam	start	roll 1	roll 2	roll 3	roll 4	final
2	1	1	2.66	0	4.83	22.14	70.72	87.51	100
2	1	2	1.80	0	10.60	33.15	75.27	83.15	100
2	1	3	1.72	0	9.78	65.76	76.36	82.61	100
2	1	4	1.93	0	6.96	54.52	83.99	93.50	100
2	1	5	1.78	0	5.25	45.19	57.14	68.80	100
2	1	6	1.94	0	5.56	37.96	63.43	75.00	100
2	1	7	1.34	0	0.78	76.56	79.69	83.59	100
2	3	8	2.33	0	0.70	31.71	48.43	59.58	100
2	3	9	2.32	0	1.22	34.03	69.79	77.78	100
2	3	10	2.19	0	5.38	30.27	51.35	63.00	100
2	3	11	2.34	0	2.51	29.15	63.82	68.68	100
2	3	12	2.16	0	4.65	16.74	76.28	83.72	100
2	3	13	2.03	0	1.10	27.03	59.56	74.07	100
2	3	14	1.56	0	1.50	2.26	2.26	3.76	100
2	3	15	1.40	0	2.63	7.89	8.42	20.53	100
4	1	1	2.49	0	19.62	92.09	98.26	98.89	100
4	1	2	2.20	0	100.00	100.00	100.00	100.00	100
4	1	3	2.02	0	10.59	95.09	97.93	97.93	100
4	1	4	2.86	0	0.68	4.21	11.05	53.30	100
4	1	5	2.54	0	93.34	99.15	99.66	100.00	100
4	2	6	2.25	0	8.67	80.10	98.39	100.00	100
4	2	7	2.40	0	2.30	69.27	92.83	99.10	100
4	2	8	2.00	0	84.67	95.86	96.11	100.00	100
4	2	9	2.18	0	2.36	58.74	92.93	94.89	100
4	2	10	2.83	0	31.65	98.31	99.22	99.87	100
4	3	11	2.08	0	43.91	94.10	98.89	99.26	100
4	3	12	2.16	0	1.78	82.81	92.69	94.07	100
4	3	13	1.83	0	10.40	80.61	93.62	99.76	100
4	3	14	2.06	0	86.99	99.74	98.98	100.00	100
4	3	15	2.10	0	15.35	90.93	98.37	100.00	100
6	1	1	2.22	0	74.12	97.59	98.87	99.20	100
6	1	2	2.00	0	100.00	100.00	100.00	100.00	100
6	1	3	2.42	0	31.08	92.54	94.34	97.24	100
6	1	4	2.63	0	0.14	41.39	87.43	98.09	100
6	1	5	2.12	0	100.00	100.00	100.00	100.00	100
6	2	6	2.25	0	34.55	84.64	98.90	98.90	100
6	2	7	2.31	0	28.16	83.70	100.00	100.00	100
6	2	8	2.14	0	100.00	100.00	100.00	100.00	100
6	2	9	2.23	0	88.50	100.00	100.00	100.00	100
6	2	10	2.24	0	100.00	100.00	100.00	100.00	100
6	3	11	2.02	0	31.15	100.00	100.00	100.00	100
6	3	12	2.26	0	76.87	95.10	96.70	97.31	100
6	3	13	1.93	0	100.00	100.00	100.00	100.00	100
6	3	14	2.13	0	97.02	100.00	100.00	100.00	100
6	3	15	1.71	0	31.53	99.70	100.00	100.00	100
8	1	1	2.24	0	100.00	100.00	100.00	100.00	100
8	1	2	2.38	0	100.00	100.00	100.00	100.00	100
8	1	3	2.57	0	100.00	100.00	100.00	100.00	100
8	1	4	2.13	0	100.00	100.00	100.00	100.00	100
8	1	5	2.50	0	30.39	98.91	99.07	100.00	100
8	2	6	2.36	0	100.00	100.00	100.00	100.00	100
8	2	7	2.59	0	100.00	100.00	100.00	100.00	100
8	2	8	1.99	0	100.00	100.00	100.00	100.00	100

8	2	9	2.11	0	100.00	100.00	100.00	100.00	100
8	2	10	2.11	0	100.00	100.00	100.00	100.00	100
8	3	11	2.06	0	85.12	85.12	100.00	100.00	100
8	3	12	2.02	0	88.01	88.18	97.71	98.06	100
8	3	13	2.07	0	100.00	100.00	100.00	100.00	100
8	3	14	2.00	0	100.00	100.00	100.00	100.00	100
8	3	15	1.79	0	100.00	100.00	100.00	100.00	100